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# DICTIONARY OF ORGANIC COMPOUNDS

## VOLUME III



# DICTIONARY OF ORGANIC COMPOUNDS<sup>9</sup>

THE CONSTITUTION AND PHYSICAL AND CHEMICAL  
PROPERTIES OF THE PRINCIPAL CARBON COM-  
POUNDS AND THEIR DERIVATIVES, TOGETHER  
WITH THE RELEVANT LITERATURE REFERENCES

VOLUME THREE

## NAPHTHACARBAZOLE — ZYGADENINE

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## TABLE OF ABBREVIATIONS

<b>A</b>	Acid ( $A_2$ , two mols of acid).	<b>I.U.</b>	International Unit.
<b>Å</b>	Angstrom unit. ( $10^{-8}$ cm.).	<b>Jap. P.</b>	Japanese Patent.
<b>Abs. EtOH</b>	Absolute alcohol.	<b>k</b>	Dissociation constant.
<b>AcOH</b>	Acetic acid.	<b>l</b>	Levorotatory.
<b>Ac<sub>2</sub>O</b>	Acetic anhydride.	<b>Liq.</b>	Liquid.
<b>AcOEt</b>	Ethyl acetate.	<b>m</b>	Meta (position).
<b>Add.</b>	Additive.	<b>Max.</b>	Maximum.
<b>Addn.</b>	Addition.	<b>Me</b>	Methyl.
<b>A.G.F.A.</b>	Aktien-Gesellschaft für Anilinfabrikation.	<b>MeOH</b>	Methyl alcohol.
<b>Alc.</b>	Alcohol, alcoholic.	<b>Me<sub>2</sub>CO</b>	Acetone.
<b>Alc. NH<sub>3</sub></b>	Alcoholic ammonia.	<b>Min.</b>	Mineral (inorganic).
<b>Alk.</b>	Alkali, alkaline.	<b>Misc.</b>	Miscible.
<b>[α]</b>	Specific rotation.	<b>M.L.B.</b>	Meister, Lucius, & Brünig.
<b>Amorph.</b>	Amorphous.	<b>mm.</b>	Millimetres.
<b>Anhyd.</b>	Anhydrous.	<b>Mod.</b>	Moderately.
<b>Aq.</b>	Aqueous.	<b>Mol.</b>	Molecule, molecular, molar.
<b>Atm.</b>	Atmosphere(s), atmospheric.	<b>M.p.</b>	Melting point.
<b>B</b>	Base ( $B_2$ , two mols of base).	<b>ms</b>	Meso (position).
<b>Badische</b>	Badische Anilin und Sodafabrik.	<b>MW</b>	Molecular weight (formula weight).
<b>Belg. P.</b>	Belgian Patent.	<b>mgm.</b>	Milligramme(s).
<b>B.D.C.</b>	British Dyestuffs Corporation.	<b>mμ</b>	Millimicron. ( $10^{-7}$ cm.).
<b>Bibl.</b>	Bibliography.	<b>n</b>	Normal (chain).
<b>B.p.</b>	Boiling point.	<b>n<sub>D</sub></b>	Refractive index (D line, etc.).
<b>C<sub>p</sub></b>	Constant pressure.	<b>NaHg</b>	Sodium amalgam.
<b>C<sub>v</sub></b>	Constant volume.	<b>NH<sub>3</sub></b>	Ammonia, aqueous ammonia.
<b>Cal.</b>	Calories.	<b>NH<sub>3</sub>·AgNO<sub>3</sub></b>	Ammoniacal silver nitrate.
<b>Can. P.</b>	Canadian Patent.	<b>o</b>	Ortho (position).
<b>Col.</b>	Colour, coloration.	<b>Ord.</b>	Ordinary.
<b>Comb.</b>	Combustion.	<b>Org.</b>	Organic.
<b>Comp.</b>	Compound.	<b>Ox.</b>	Oxidise, oxidation.
<b>Conc.</b>	Concentrated.	<b>p</b>	Para (position).
<b>Corr.</b>	Corrected.	<b>P</b>	Patent.
<b>Crit.</b>	Critical.	<b>Part.</b>	Partly, partial.
<b>Cryst.</b>	Crystals, crystalline, crystallise.	<b>Pet. ether</b>	Petroleum ether.
<b>(COOH)<sub>2</sub></b>	Oxalic acid.	<b>PhNO<sub>2</sub></b>	Nitrobenzene.
<b>(CH<sub>2</sub>COOH)<sub>2</sub></b>	Succinic acid.	<b>PhOH</b>	Phenol.
<b>D</b>	Density.	<b>Ppd.</b>	Precipitated.
<b>d</b>	Dextrorotatory.	<b>Ppt.</b>	Precipitate.
<b>dl</b>	Racemic. Optically inactive by external compensation.	<b>Pptn.</b>	Precipitation.
<b>Decomp.</b>	Decomposed, decomposition.	<b>Prac.</b>	Practically.
<b>Deriv.</b>	Derivative.	<b>Press.</b>	Pressure(s).
<b>Dil.</b>	Dilute, dilution.	<b>ψ</b>	Pseudo.
<b>Diss.</b>	Dissolves, dissolved.	<b>Py</b>	Pyridine.
<b>Dist.</b>	Distil, distillation.	<b>r</b>	Racemic.
<b>D.R.P.</b>	German Patent.	<b>Red.</b>	Reduce, reduction.
<b>E.P.</b>	English (British) Patent.	<b>Ref.</b>	Reference.
<b>Et</b>	Ethyl.	<b>Russ.P.</b>	Russian Patent.
<b>Et<sub>2</sub>O</b>	Ether (diethyl ether).	<b>S.C.I.</b>	Société pour l'industrie chimique à Basle.
<b>EtOH</b>	Ethyl alcohol.	<b>Sec.</b>	Secondary.
<b>Fluor.</b>	Fluoresces, fluorescence.	<b>Sol.</b>	Soluble, solution.
<b>F.p.</b>	Freezing point.	<b>Spar.</b>	Sparingly.
<b>F.P.</b>	French Patent.	<b>Sp. gr.</b>	Specific gravity.
<b>Form.</b>	Formation.	<b>Sp. heat</b>	Specific heat.
<b>γ</b>	$10^{-3}$ gm. or $10^{-3}$ mgm. (microgrammes).	<b>Suppl.</b>	Supplement.
<b>gm.</b>	Gramme(s).	<b>Sym.</b>	Symmetrical.
<b>Hyd.</b>	Hydrolyses, hydrolysed, hydrolysis.	<b>Temp.</b>	Temperature(s).
<b>i</b>	Optically inactive by internal compensation.	<b>Tert.</b>	Tertiary.
<b>I.C.I.</b>	Imperial Chemical Industries.	<b>Undecomp.</b>	Undecomposed.
<b>I.G.</b>	Interessen Gemeinschaft Farbenindustrie Aktien-Gesellschaft.	<b>Unsym.</b>	Unsymmetrical.
<b>Insol.</b>	Insoluble.	<b>UV.</b>	Ultraviolet.
		<b>Vac.</b>	Vacuum.
		<b>Vap.</b>	Vaporisation.
		<b>Vol.</b>	Volume.

# JOURNAL ABBREVIATIONS

Journals not listed here are given their full titles in the text.

<i>Acta Phytochim.</i>	Acta Phytochimica (Japan).	<i>Chem. Trade J.</i>	Chemical Trade Journal (and Chemical Engineer).
<i>Am. Chem. J.</i>	American Chemical Journal.	<i>Chem. Umschau</i>	Chemische Umschau (auf dem Gebiete der Fette, Oele, Wachse, und Harze). Now Fettchemische Umschau.
<i>Am. J. Pharm.</i>	American Journal of Pharmacy.	<i>Chem. Weekblad</i>	Chemisch Weekblad.
<i>Am. J. Sci.</i>	American Journal of Science.	<i>Chem. Zentr.</i>	Chemisches Zentralblatt.
<i>Anales soc. españ. fis. quim.</i>	Anales de la sociedad española de física y química.	<i>Chem.-Ztg.</i>	Chemiker-Zeitung.
<i>Angew. Chem.</i>	Angewandte Chemie.	<i>Compt. rend.</i>	Comptes rendus (hebdomadaires des séances de l'académie des sciences).
<i>Ann.</i>	Annalen der Chemie.	<i>Compt. rend. acad. sci. U.R.S.S.</i>	Comptes rendus de l'Académie des Sciences de l'U.R.S.S.
<i>Ann. chim.</i>	Annales de chimie.	<i>Compt. rend. soc. biol.</i>	Comptes rendus des séances de la société de biologie.
<i>Ann. chim. applicata</i>	Annali di chimica applicata.	<i>Dinglers polytech. J.</i>	Dinglers polytechnisches Journal.
<i>Ann. chim. phys.</i>	Annales de chimie et de physique.	<i>Fettchem. Umschau</i>	Fettchemische Umschau.
<i>Ann. phys.</i>	Annales de physique.	<i>Gazz. chim. ital.</i>	Gazzetta chimica italiana.
<i>Ann. Physik</i>	Annalen der Physik.	<i>Giorn. chim. applicata</i>	Giornale di chimica applicata.
<i>Ann. Rev. Biochem.</i>	Annual Review of Biochemistry.	<i>Giorn. chim. ind.</i>	Giornale di chimica industriale.
<i>Arch. Pharm.</i>	Archiv der Pharmazie (und Berichte der deutschen pharmazeutischen Gesellschaft).	<i>Giorn. chim. ind. applicata</i>	Giornale di chimica industriale ed applicata.
<i>Arkiv Kemi, Mineral. Geol.</i>	Arkiv för Kemi, Mineralogi och Geologi.	<i>Helv. Chim. Acta</i>	Helvetica Chimica Acta.
<i>Atti accad. Lincei</i>	Atti della reale accademia nazionale dei Lincei.	<i>Ind. Eng. Chem.</i>	Industrial and Engineering Chemistry.
<i>Ber.</i>	Berichte der deutschen chemischen Gesellschaft.	<i>Jahresber. Fortschr. Chem.</i>	Jahresbericht über die Fortschritte der Chemie.
<i>Ber. deut. pharm. Ges.</i>	Berichte der deutschen pharmazeutischen Gesellschaft.	<i>J. Am. Chem. Soc.</i>	Journal of the American Chemical Society.
<i>Ber. ges. Physiol. exp. Pharmakol.</i>	Berichte über die gesamte Physiologie und experimentelle Pharmakologie.	<i>J. Am. Pharm. Assocn.</i>	Journal of the American Pharmaceutical Association.
<i>Biochem. J.</i>	Biochemical Journal.	<i>J. Applied Chem., U.S.S.R.</i>	Journal of Applied Chemistry, U.S.S.R.
<i>Biochem. Z.</i>	Biochemische Zeitschrift.	<i>Japan. J. Chem.</i>	Japanese Journal of Chemistry.
<i>Biol. Zentr.</i>	Biologisches Zentralblatt.	<i>J. Bact.</i>	Journal of Bacteriology.
<i>Brit. Chem. Abstracts</i>	British Chemical Abstracts.	<i>J. Biochem. Japan.</i>	Journal of Biochemistry of Japan.
<i>Bull. Chem. Soc. Japan</i>	Bulletin of the Chemical Society of Japan.	<i>J. Biol. Chem.</i>	Journal of Biological Chemistry.
<i>Bull. Imp. Inst.</i>	Bulletin of the Imperial Institute.	<i>J. Chem. Education</i>	Journal of Chemical Education.
<i>Bull. Inst. Phys. Chem. Research (Tokyo).</i>	Bulletin of the Institute of Physical and Chemical Research, Toyko.	<i>J. Chem. Ind. Japan</i>	Journal of Chemical Industry (Japan). Now J. Soc. Chem. Ind. Japan.
<i>Bull. sci. acad. roy. Belg.</i>	Bulletin de la classe des sciences, academie royale de Belgique.	<i>J. Chem. Physics</i>	Journal of Chemical Physics.
<i>Bull. sci. pharmacol.</i>	Bulletin des sciences pharmacologiques.	<i>J. Chem. Soc.</i>	Journal of the Chemical Society (London).
<i>Bull. soc. chim.</i>	Bulletin de la société chimique de France.	<i>J. Chem. Soc. Abstracts</i>	Abstracts of the Chemical Society (London).
<i>Bull. soc. chim. Belg.</i>	Bulletin de la société chimique de Belgique.	<i>J. Chem. Soc. Japan</i>	Journal of the Chemical Society of Japan.
<i>Bull. soc. chim. biol.</i>	Bulletin de la société de chimie biologique.	<i>J. chim. phys.</i>	Journal de chimie physique.
<i>Can. Chem. Met.</i>	Canadian Chemistry and Metallurgy.	<i>J. Chinese Chem. Soc.</i>	Journal of the Chinese Chemical Society.
<i>Can. J. Research</i>	Canadian Journal of Research.	<i>J. Gen. Chem. U.S.S.R.</i>	Journal of General Chemistry, U.S.S.R.
<i>Chem. Abstracts</i>	Chemical Abstracts (of the American Chemical Society).	<i>J. Indian Chem. Soc.</i>	Journal of the Indian Chemical Society.
<i>Chem. Ind.</i>	Die Chemische Industrie.	<i>J. Indian Inst. Sci.</i>	Journal of the Indian Institute of Science.
<i>Chem. Met. Eng.</i>	Chemical and Metallurgical Engineering.	<i>J. Org. Chem.</i>	Journal of Organic Chemistry.
<i>Chem. News</i>	Chemical News (and Journal of Industrial Science).	<i>J. Pharmacol.</i>	Journal of Pharmacology and Experimental Therapeutics.
<i>Chem.-Tech. Rundschau</i>	Chemische-Technische Rundschau.	<i>J. pharm. Belg.</i>	Journal de pharmacie de Belgique.

<i>J. pharm. chim.</i>	Journal de pharmacie et de chimie.	<i>Proc. Imper. Acad., Tokyo</i>	Proceedings of the Imperial Academy, Tokyo.
<i>J. Pharm. Soc. Japan</i>	Journal of the Pharmaceutical Society (Japan).	<i>Quart. J. Indian Chem. Soc.</i>	Quarterly Journal of the Indian Chemical Society.
<i>J. Phys. Chem.</i>	Journal of Physical Chemistry.	<i>Quart. J. Pharm. Pharmacol.</i>	Quarterly Journal of Pharmacy and Pharmacology.
<i>J. prakt. Chem.</i>	Journal für praktische Chemie.	<i>Rec. trav. chim.</i>	Recueil des travaux chimiques des Pays-Bas.
<i>J. Proc. Roy. Soc. N.S. Wales</i>	Journal and Proceedings of the Royal Society of New South Wales.	<i>Rev. chim. ind.</i>	Revue de chimie industrielle.
<i>J. Russ. Phys.-Chem. Soc.</i>	Journal of the Russian Physical-Chemical Society.	<i>Rev. prod. chim.</i>	Revue des produits chimiques.
<i>J. Soc. Chem. Ind.</i>	Journal of the Society of Chemical Industry.	<i>Sci. Papers Inst. Phys. Chem. Research, Tokyo</i>	Scientific Papers of the Institute of Physical and Chemical Research (Tokyo).
<i>J. Soc. Chem. Ind. Japan</i>	Journal of the Society of Chemical Industry (Japan).	<i>Sci. reps. Natl. Tsinghua Univ.</i>	Science Reports of the National Tsinghua University.
<i>J. Soc. Dyers Colourists</i>	Journal of the Society of Dyers and Colourists.	<i>Sci. reps. Natl. Univ. Peking</i>	Science Reports of the National University of Peking.
<i>Mem. Coll. Sci., Kyoto Imp. Univ. Monatsh.</i>	Memoirs of the College of Science, Kyoto Imperial University. Monatshefte für Chemie und verwandte Teile anderer Wissenschaften.	<i>Sitzb. Akad. Wiss. Wien</i>	Sitzungsberichte Akademie der Wissenschaften in Wien.
<i>Naturwiss.</i>	Naturwissenschaften.	<i>Trans. Faraday Soc.</i>	Transactions of the Faraday Society.
<i>Org. Chem. Ind. U.S.S.R.</i>	Promishlennosti Organitscheskoi Chimii, U.S.S.R.	<i>Trans. Roy. Soc. Canada.</i>	Transactions of the Royal Society of Canada.
<i>Pharm. J.</i>	Pharmaceutical Journal and Pharmacist.	<i>Z. anal. Chem.</i>	Zeitschrift für analytische Chemie.
<i>Pharm. Ztg.</i>	Die deutsche Pharmazeutische Zeitung.	<i>Z. angew. Chem.</i>	Zeitschrift für angewandte Chemie. Now Angewandte Chemie.
<i>Pharm. Zentralhalle.</i>	Pharmazeutische Zentralhalle.	<i>Z. anorg. allgem. Chem.</i>	Zeitschrift für anorganische und allgemeine Chemie.
<i>Phil. Mag.</i>	Philosophical Magazine and Journal of Science.	<i>Z. Chem.</i>	Zeitschrift für Chemie.
<i>Proc. Acad. Sci., Amsterdam</i>	Proceedings of the Royal Academy of Sciences of Amsterdam.	<i>Z. Elektrochem.</i>	Zeitschrift für Elektrochemie und angewandte physikalische Chemie.
<i>Proc. Chem. Soc.</i>	Proceedings of the Chemical Society (London).	<i>Z. ges Naturwiss.</i>	Zeitschrift für die gesamte Naturwissenschaft.
<i>Proc. Roy. Soc.</i>	Proceedings of the Royal Society (London).	<i>Z. physik. Chem.</i>	Zeitschrift für physikalische Chemie.
		<i>Z. physiol. Chem.</i>	Zeitschrift für physiologische Chemie (Hoppe-Seyler).

## LIST OF SUBSTITUENTS

In the following table is given a list of the principal substituent groups as they are used in the dictionary.

1 —F	Fluoro	17 —SO <sub>2</sub> H	Sulpho
2 —Cl	Chloro	18 —NH <sub>2</sub>	Amino
3 —Br	Bromo	19 —NH·C <sub>6</sub> H <sub>5</sub>	Anilino, Phenylimino
4 —I	Iodo	20 —NH·C <sub>6</sub> H <sub>4</sub> ·CH <sub>3</sub>	Toluidino
5 —NO	Nitroso	21 —NH·CO·NH <sub>2</sub>	Ureido
6 —NO <sub>2</sub>	Nitro	22 —NH·C(NH) <sub>2</sub> ·NH <sub>2</sub>	Guanidino
7 —N=N→N	Azido, Triazo	23 —NH·OH	Hydroxylamino
8 —OH	Hydroxy (followed by —OCH <sub>3</sub> , Methoxy, —OC <sub>2</sub> H <sub>5</sub> , Ethoxy, —O·CH <sub>2</sub> ·O— methylenedioxy, —OC <sub>6</sub> H <sub>5</sub> , Phenoxy, —O·CO·CH <sub>3</sub> , Acetoxy, etc. in the order of the group attached to the oxygen)	24 —NH·NH <sub>2</sub>	Hydrazino
9 —SH	Mercapto	25 —NH·NH—	Hydrazo
10 —SO	Thionyl	26 —N·N—	Azo
11 —SO <sub>2</sub>	Sulphonyl	27 ·N≡N·X	Diazonium, Diazo (X=OH, Cl, etc.)
12 —SCN	Thiocyano	28 —N=N—   O	Azoxy
13 =O (in C—CO—C)	Keto	29 —As·As—	Arseno
14 >NH	Imino	30 —NH·N·N— (open)	Diazoamino
15 =N·OH	Isonitroso, Oximino	31 —NH·N·N— (cyclic)	Azimino
16 —S—	Thio	32 —CH <sub>3</sub>	Methyl
		33 —CH <sub>2</sub> OH	Hydroxymethyl, Methylol
		34 —C <sub>2</sub> H <sub>5</sub>	Ethyl

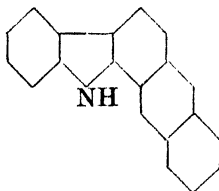


35	$-\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2$	<i>n</i> -Propyl	99	$-\text{CH}_2\cdot[\text{CH}_2]_6\cdot\text{CH}_2-$	Heptamethylene
36	$-\text{CH}(\text{CH}_3)_2$	Isopropyl	100	$-\text{CH}_2\cdot[\text{CH}_2]_6\cdot\text{CH}_2-$	Octamethylene
37	$-\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2$	<i>n</i> -Butyl	101	$-\text{CH}\cdot\text{CH}-$	Vinylene
38	$-\text{CH}_2\cdot\text{CH}(\text{CH}_3)_2$	Isobutyl	102	$-\text{C}_6\text{H}_5-$	Phenylene
39	$-\text{C}(\text{CH}_3)_3$	<i>tert</i> -Butyl	103	$-\text{C}_6\text{H}_5(\text{CH}_2)-$	Tolylene
40	$-\text{CH}_2\cdot[\text{CH}_2]_3\cdot\text{CH}_2$	<i>n</i> -Amyl	104	$-\text{CH}_2-$	Methylene
41	$-\text{CH}(\text{C}_2\text{H}_5)_2$	<i>sec</i> - <i>n</i> -Amyl	105	$=\text{CH}\cdot\text{CH}_2$	Ethylidene
42	$-\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}(\text{CH}_3)_2$	Isoamyl	106	$=\text{CH}\cdot\text{CH}_2\cdot\text{CH}_2$	Propylidene
43	$-\text{CH}_2\cdot\text{CH}\begin{matrix} \text{CH}_3 \\ \diagup \\ \text{C}_2\text{H}_5 \end{matrix}$	active Amyl	107	$=\text{C}(\text{CH}_3)_2$	Isopropylidene
44	$-\text{C}\begin{matrix} \text{CH}_3 \\ \diagup \\ \text{C}_2\text{H}_5 \\ \diagdown \\ \text{CH}_3 \end{matrix}$	<i>tert</i> -Amyl	108	$=\text{CH}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2$	Butylidene
45	$-\text{CH}_2\cdot[\text{CH}_2]_4\cdot\text{CH}_2$	<i>n</i> -Hexyl	109	$=\text{CH}\cdot\text{CH}(\text{CH}_3)_2$	Isobutylidene
46	$-\text{CH}_2\cdot[\text{CH}_2]_4\cdot\text{CH}(\text{CH}_3)_2$	Isohexyl	110	$\text{H}_2\text{C}\begin{matrix} \text{CH}_2-\text{CH}_2 \\ \diagup \quad \diagdown \\ \text{CH}_2-\text{CH}_2 \end{matrix}\text{C}=\text{C}$	Cyclohexylidene
47	$-\text{CH}_2\cdot[\text{CH}_2]_5\cdot\text{CH}_2$	<i>n</i> -Heptyl, Oenanthylyl	111	$=\text{C}\cdot\text{CH}_2$	Vinylidene
48	$-\text{CH}_2\cdot[\text{CH}_2]_5\cdot\text{CH}(\text{CH}_3)_2$	Isoheptyl	112	$=\text{CH}\cdot\text{CH}\cdot\text{CH}_2$	Allylidene
49	$-\text{CH}_2\cdot[\text{CH}_2]_6\cdot\text{CH}_2$	Octyl, Caprylyl	113	$\text{CH}_2\cdot\text{CH}\cdot\text{CH}\cdot\text{CH}=\text{CH}$	Crotylidene
50	$-\text{CH}_2\cdot[\text{CH}_2]_7\cdot\text{CH}_2$	Nonyl	114	$=\text{CH}\cdot\text{C}_6\text{H}_5$	Benzylidene
51	$-\text{CH}_2\cdot[\text{CH}_2]_8\cdot\text{CH}_2$	Decyl	115	$=\text{CH}\cdot\text{C}_6\text{H}_4\cdot\text{OH} (-o)$	Salicylidene
52	$-\text{CH}_2\cdot[\text{CH}_2]_9\cdot\text{CH}_2$	Undecyl	116	$=\text{CH}\cdot\text{C}_6\text{H}_4\cdot\text{OCH}_3 (-p)$	Anisylidene
53	$-\text{CH}_2\cdot[\text{CH}_2]_{10}\cdot\text{CH}_2$	Dodecyl	117	$=\text{CH}\cdot\text{C}_6\text{H}_4\cdot\text{CH}(\text{CH}_3)_2 (-p)$	Cuminyldiene
54	$-\text{CH}_2\cdot[\text{CH}_2]_{11}\cdot\text{CH}_2$	Tridecyl	118	$=\text{CH}\cdot\text{CH}\cdot\text{CH}\cdot\text{C}_6\text{H}_5$	Cinnamylidene
55	$-\text{CH}_2\cdot[\text{CH}_2]_{12}\cdot\text{CH}_2$	Tetradecyl	119	$-\text{CH}_2\cdot\text{CO}\cdot\text{CH}_2$	Acetonyl
56	$-\text{CH}_2\cdot[\text{CH}_2]_{13}\cdot\text{CH}_2$	Pentadecyl	120	$-\text{CH}_2\cdot\text{CO}\cdot\text{C}_6\text{H}_5$	Phenacyl
57	$-\text{CH}_2\cdot[\text{CH}_2]_{14}\cdot\text{CH}_2$	Cetyl, Hexadecyl	121	$-\text{CH}_2\cdot\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2$	Tolacyl
58	$-\text{CH}_2\cdot[\text{CH}_2]_{15}\cdot\text{CH}_2$	Heptadecyl	122	$\text{C}_6\text{H}_5\cdot\text{CH}\cdot\text{CO}\cdot\text{C}_6\text{H}_5$	Desyl
59	$-\text{CH}_2\cdot[\text{CH}_2]_{16}\cdot\text{CH}_2$	Octadecyl	123	$-\text{CH}\cdot\text{O}$	Aldehydo, Formyl
60	$-\text{CH}_2\cdot[\text{CH}_2]_{17}\cdot\text{CH}_2$	Eicosyl	124	$=\text{CH}$	Methinyl
61	$-\text{CH}_2\cdot[\text{CH}_2]_{18}\cdot\text{CH}_2$	Ceryl	125	$-\text{CO}\cdot\text{CH}_2$	Acetyl, Aceto
62	$-\text{CH}_2\cdot[\text{CH}_2]_{25}\cdot\text{CH}_2$	Myricyl, Melissyl	126	$-\text{CO}\cdot\text{CH}_2\cdot\text{CH}_2$	Propionyl
63	$-\text{CH}\begin{matrix} \text{CH}_2 \\ \diagup \\ \text{CH}_2 \end{matrix}$ Cyclopropyl (followed by Cyclobutyl, Cyclopentyl, Cyclohexyl, Cycloheptyl (Suberyl) in that order)		127	$-\text{CO}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2$	Butyryl
64	$-\text{CH}\cdot\text{CH}_2$	Vinyl	128	$-\text{CO}\cdot\text{CH}(\text{CH}_3)_2$	Isobutyryl
65	$-\text{CH}\cdot\text{CH}\cdot\text{CH}_2$	Propenyl	129	$-\text{CO}\cdot\text{CH}_2\cdot[\text{CH}_2]_3\cdot\text{CH}_2$	Valeryl
66	$-\text{C}(\text{CH}_3)_2\cdot\text{CH}_2$	Isopropenyl	130	$-\text{CO}\cdot\text{CH}_2\cdot\text{CH}(\text{CH}_3)_2$	Isovaleryl
67	$-\text{CH}_2\cdot\text{CH}\cdot\text{CH}_2$	Allyl	131	$-\text{CO}\cdot\text{CH}_2\cdot[\text{CH}_2]_4\cdot\text{CH}_2$	Caproyl
68	$-\text{CH}_2\cdot\text{CH}\cdot\text{CH}_2\cdot\text{CH}_2$	$\alpha$ -Butenyl	132	$-\text{CO}\cdot\text{CH}_2\cdot[\text{CH}_2]_5\cdot\text{CH}_2$	Palmityl
69	$-\text{CH}_2\cdot\text{CH}\cdot\text{CH}\cdot\text{CH}_2$	$\beta$ -Butenyl, Crotyl	133	$-\text{CO}\cdot\text{CH}_2\cdot[\text{CH}_2]_{15}\cdot\text{CH}_2$	Stearyl
70	$-\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}\cdot\text{CH}_2$	$\gamma$ -Butenyl, Allylomethyl	134	$-\text{CO}\cdot[\text{CH}_2]_7\cdot\text{CH}_2\cdot\text{CH}\cdot[\text{CH}_2]_7\cdot\text{CH}_2$	Oleoyl
71	$-\text{CH}_2\cdot[\text{CH}_2]_7\cdot\text{CH}\cdot\text{CH}\cdot[\text{CH}_2]_7\cdot\text{CH}_2$	Octadecenyl	135	$-\text{CO}\cdot\text{C}_6\text{H}_5$	Benzoyl
72	$-\text{C}\cdot\text{CH}$	Acetylenyl, Ethinyl	136	$-\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{OH} (-o)$	Salicyloyl
73	$-\text{CH}_2\cdot\text{C}\cdot\text{CH}$	Propargyl	137	$-\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{OCH}_3 (-p)$	Anisoyl
74	$-\text{C}_6\text{H}_5$	Phenyl	138	$-\text{CO}\cdot\text{CH}_2\cdot\text{C}_6\text{H}_5$	Phenylacetyl
75	$-\text{C}_6\text{H}_4\cdot\text{CH}_2$	Tolyl	139	$-\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2$	Toluylyl
76	$-\text{CH}_2\cdot\text{C}_6\text{H}_5$	Benzyl	140	$-\text{CO}\cdot\text{CH}\cdot\text{CH}\cdot\text{C}_6\text{H}_5$	Cinnamoyl
77	$-\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{OH} (-o)$	Salicyl	141	$-\text{CO}\cdot\text{C}_{10}\text{H}_7$	Naphthoyl
78	$-\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{OCH}_3 (-p)$	Anisyl	142	$-\text{CO}\cdot\text{CO}-$	Oxalyl
79	$-\text{CH}_2\cdot\text{CH}_2\cdot\text{C}_6\text{H}_5$	Phenylethyl	143	$-\text{CO}\cdot\text{CH}_2\cdot\text{CO}-$	Malonyl
80	$-\text{CH}_2\cdot\text{C}_6\text{H}_4\cdot\text{CH}_2$	Xylyl	144	$-\text{CO}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CO}-$	Succinyl
81	$-\text{C}_6\text{H}_4\cdot\text{CH}(\text{CH}_3)_2$	Cumyl	145	$-\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{CO}-$	
82	$-\text{C}_6\text{H}_3(\text{CH}_3)_2 (1:2:4)$	$\psi$ -Cumyl			Phthaloyl, Isophthaloyl, Terephthaloyl
83	$-\text{C}_6\text{H}_3(\text{CH}_3)_3 (1:3:5)$	Mesityl	146	$-\text{COOH} (-\text{CO}\cdot\text{OCH}_3, -\text{CO}\cdot\text{OC}_2\text{H}_5, \text{etc.})$	Carboxy, (Carbomethoxy, Carbethoxy, etc.)
84	$-\text{CH}_2\cdot\text{CH}\cdot\text{C}_6\text{H}_5$	Styryl	147	$-\text{CO}\cdot\text{NH}_2$	Carbamyl
85	$-\text{CH}_2\cdot\text{CH}\cdot\text{CH}\cdot\text{C}_6\text{H}_5$	Cinnamyl	148	$>\text{CO}$	Carbonyl
86	$-\text{C}_{10}\text{H}_7$	Naphthyl	149	$-\text{C}(\text{NH})\cdot\text{NH}_2$	Guanyl
87	$-\text{C}_6\text{H}_4\cdot\text{C}_6\text{H}_5$	Diphenyl, Xenyl	150	$-\text{CN}$	Cyano
88	$-\text{CH}(\text{C}_6\text{H}_5)_2$	Benzhydryl, Diphenylmethyl	151	$-\text{CO}\cdot\text{CH}_2\cdot\text{NH}_2$	Glycyl
89	$-\text{C}_{14}\text{H}_9$	Anthryl, anthranlyl	152	$-\text{CO}\cdot\text{CH}(\text{NH}_2)\cdot\text{CH}_2$	$\alpha$ -Alanlyl
90	$-\text{C}_{15}\text{H}_{11}$	Phenanthryl	153	$-\text{CO}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{NH}_2$	$\beta$ -Alanlyl
91	$-\text{C}(\text{C}_6\text{H}_5)_3$	Triphenylmethyl	154	$-\text{CO}\cdot\text{CH}(\text{NH}_2)\cdot\text{CH}(\text{CH}_3)_2$	Valyl
92	$-\text{CH}_2\cdot\text{CH}_2-$	Ethylene, Dimethylene	155	$-\text{CO}\cdot\text{CH}(\text{NH}_2)\cdot\text{CH}_2\cdot\text{CH}(\text{CH}_3)_2$	Leucyl
93	$-\text{CH}(\text{CH}_3)_2-$	Propylene	156	$-\text{CO}\cdot\text{CH}_2\cdot\text{NH}\cdot\text{CO}\cdot\text{C}_6\text{H}_5$	Hippuryl
94	$-\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2-$	Trimethylene	157	$-\text{C}_6\text{H}_5\cdot\text{O}$	Furyl
95	$-\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2-$	Tetramethylene	158	$-\text{C}_6\text{H}_5\cdot\text{S}$	Thienyl
96	$-\text{C}(\text{CH}_3)_3-$	Isobutylene	159	$-\text{CH}_2\cdot\text{C}_6\text{H}_5\cdot\text{O}$	Furfuryl
97	$-\text{CH}_2\cdot[\text{CH}_2]_4\cdot\text{CH}_2-$	Pentamethylene	160	$=\text{CH}\cdot\text{C}_6\text{H}_5\cdot\text{O}$	Furfurylidene
98	$-\text{CH}_2\cdot[\text{CH}_2]_6\cdot\text{CH}_2-$	Hexamethylene	161	$-\text{CO}\cdot\text{C}_6\text{H}_5\cdot\text{O}$	Furoyl, Pyromucyl
			162	$-\text{C}_6\text{H}_5\cdot\text{NH}$	Pyrryl
			163	$-\text{C}_6\text{H}_5\cdot\text{N}$	Pyridyl

# DICTIONARY OF ORGANIC COMPOUNDS

## N

### 2 : 3-Naphthacarbazole



$C_{20}H_{13}N$

MW, 267

Yellow leaflets with greenish fluor. M.p.  $325^{\circ}$ . Very spar. sol. org. solvents. Conc.  $H_2SO_4 \rightarrow$  deep blue col.

Braun, Bayer, *Ann.*, 1929, **472**, 97, 101.

### Naphthacene (2 : 3-Benzanthracene)



$C_{18}H_{12}$

MW, 228

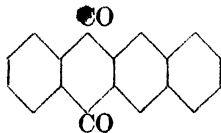
Orange-red leaflets from xylene. M.p.  $341^{\circ}$  ( $335-6^{\circ}$ ,  $331^{\circ}$ ). Sublimes  $\rightarrow$  greenish-yellow vapour. Sol. conc.  $H_2SO_4 \rightarrow$  dull green col. Insol.  $C_6H_6$ . Fuming  $HNO_3 \rightarrow$  naphthacene-quinone.

Clar, *Ber.*, 1932, **65**, 517.

Dziewoński, Ritt, *Chem. Abstracts*, 1928, **22**, 2561.

Deichler, Weizmann, *Ber.*, 1903, **36**, 552.

**Naphthacenequinone** (2 : 3-Benzanthraquinone, lin-benzanthraquinone, 2 : 3-phthaloylnaphthalene)



$C_{18}H_{10}O_2$

MW, 258

Cryst. from  $PhNO_2$  in yellow needles. M.p.  $294^{\circ}$  ( $285^{\circ}$ ). Sublimes. Sol. conc.  $H_2SO_4 \rightarrow$  red-violet col. Spar. sol. hot  $C_6H_6$ , hot  $Me_2CO$ . Very spar. sol. AcOH.  $Sn + AcOH \rightarrow$  yellow needles of the anthrone, m.p.  $196^{\circ}$ , sol. conc.  $H_2SO_4$  to bright red sol.  $Sn + Ac_2O + AcONa$

Dict. of Org. Comp.—III.

$\rightarrow$  orange needles of naphthacenehydroquinone-diacetate, m.p.  $269^{\circ}$ .

Fieser, *J. Am. Chem. Soc.*, 1931, **53**, 2336.  
Waldmann, Mathiowetz, *Ber.*, 1931, **64**, 1713.

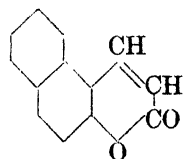
### Naphthacetin.

See under 4-Amino-1-naphthol.

### Naphthacetol.

See under 4-Amino-1-naphthol.

$\alpha$ : $\beta$ -Naphthacoumarin (5:6-Benzcoumarin)



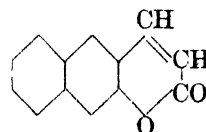
$C_{13}H_8O_2$

MW, 196

Bright yellow needles from EtOH.Aq. M.p.  $118^{\circ}$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH. Spar. sol. hot  $H_2O \rightarrow$  bluish fluor.

Dey, Rao, Sankaranarayanan, *J. Indian Chem. Soc.*, 1932, **9**, 71.

$\beta$ : $\beta$ -Naphthacoumarin (6:7-Benzcoumarin)



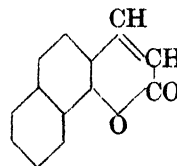
$C_{13}H_8O_2$

MW, 196

Pale yellowish cryst. from  $CHCl_3$ -pet. ether. M.p.  $163-4^{\circ}$ .

Boehm, Profft, *Arch. Pharm.*, 1931, **269**, 25 (*Chem. Zentr.*, 1931, I, 1922).

$\beta$ : $\alpha$ -Naphthacoumarin (7:8-Benzcoumarin)



$C_{13}H_8O_2$

MW, 196

## Naphthacridine

Pale yellow needles from EtOH. M.p. 141–2° (138°). Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, AcOH. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Greenish-yellow sol. in conc. H<sub>2</sub>SO<sub>4</sub> → blue fluor.

Dey, Rao, Sankaranarayanan, *J. Indian Chem. Soc.*, 1932, **9**, 71.

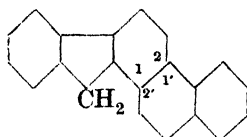
Bezdzik, Friedländer, *Monatsh.*, 1909, **30**, 280.

See also previous reference.

### Naphthacridine.

See Chrysidine.

### 2' : 1'-Naphtha-1 : 2-fluorene



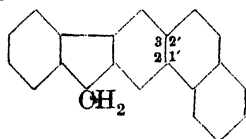
C<sub>21</sub>H<sub>14</sub>

MW, 266

Colourless leaflets from xylene. M.p. 327–8°. Forms add. comp., m.p. 249–51°, with 2 : 7-dinitroanthraquinone.

Cook et al., *J. Chem. Soc.*, 1934, 1737.

### 1' : 2'-Naphtha-2 : 3-fluorene



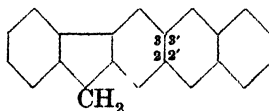
C<sub>21</sub>H<sub>14</sub>

MW, 266

Colourless leaflets from C<sub>6</sub>H<sub>6</sub>-EtOH. M.p. 226°.

Cook et al., *J. Chem. Soc.*, 1935, 1323.

### 2' : 3'-Naphtha-2 : 3-fluorene (lin-Naphtha-fluorene)



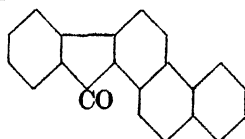
C<sub>21</sub>H<sub>14</sub>

MW, 266

Cryst. from toluene. M.p. 317°. Sol. C<sub>6</sub>H<sub>6</sub> → bright blue fluor. Sol. conc. H<sub>2</sub>SO<sub>4</sub> → bright green fluor.

Barnett, Goodway, Watson, *Ber.*, 1933, **66**, 1890.

### 2' : 1'-Naphtha-1 : 2-fluorenone



C<sub>21</sub>H<sub>12</sub>O

MW, 280

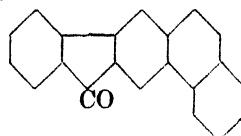
## 2

## β-Naphthafurandione

Reddish-orange needles from AcOH. M.p. 207–8°.

Cook et al., *J. Chem. Soc.*, 1934, 1737.

### 1' : 2'-Naphtha-2 : 3-fluorenone



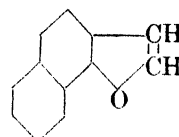
C<sub>21</sub>H<sub>12</sub>O

MW, 280

Reddish-brown needles. M.p. 215°. Sol. in conc. H<sub>2</sub>SO<sub>4</sub> → magenta col.

Cook et al., *J. Chem. Soc.*, 1935, 1323.

### α-Naphthafuran (6 : 7-Benzcoumarone)



C<sub>12</sub>H<sub>8</sub>O

MW, 168

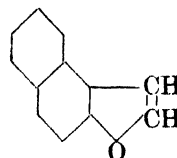
Pale yellow oil. M.p. –7°. B.p. 282–4°/755 mm. D<sub>4</sub><sup>14</sup> 1.1504. n<sub>D</sub><sup>16</sup> 1.634. Sol. conc. H<sub>2</sub>SO<sub>4</sub> → yellowish-green col., on warming → blue → violet fluor

Picrate : reddish-yellow needles. M.p. 113°.

Stoermer, *Ann.*, 1900, **312**, 310.

Boes, *Chem. Zentr.*, 1902, I, 1356.

### β-Naphthafuran (4 : 5-Benzcoumarone)



C<sub>12</sub>H<sub>8</sub>O

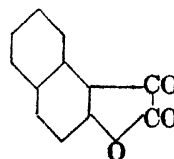
MW, 168

Needles. M.p. 60–1° (65°). B.p. 284–6° (280°). Sol. conc. H<sub>2</sub>SO<sub>4</sub> → yellowish-green col., on warming → pale violet → dirty bluish-green → brownish-violet fluor.

Picrate : red needles. M.p. 141°.

See previous references.

### β-Naphthafurandione (4 : 5-Benzcoumarandione)



C<sub>12</sub>H<sub>6</sub>O<sub>3</sub>

MW, 198

Orange-yellow needles from AcOH or C<sub>6</sub>H<sub>6</sub>.

M.p. 182° decomp. Sol. EtOH. Spar. sol. H<sub>2</sub>O, pet. ether. The blood-red sol. in conc. H<sub>2</sub>SO<sub>4</sub> turns colourless on warming.

2-Anil : m.p. 126-7°.

2 : 3-Phenazine ; m.p. 286-7°.

3-Phenylhydrazone : m.p. 226-7°.

3-Semicarbazone : m.p. 240-1°.

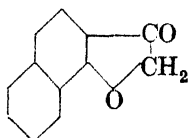
Picrate : m.p. 109°.

Giua, Francis, *Gazz. chim. ital.*, 1924, **54**, 509.

Passerini, *ibid.*, 184.

Staudinger, *Swiss Ps.*, 92,688, 93,486, (*Chem. Abstracts*, 1924, **18**, 989).

**$\alpha$ -Naphthafuranone-3** (6 : 7-Benzcoumaranone-3)



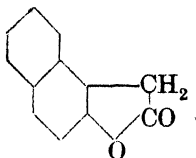
C<sub>12</sub>H<sub>8</sub>O<sub>2</sub> MW, 184

Pale yellow needles. M.p. 119°. Yellow sol. in conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  green fluor. Fehling's  $\rightarrow$  deep purple col.

2-Benzylidene deriv. : m.p. 130°.

Ingham, Stephen, Timpe, *J. Chem. Soc.*, 1931, 895.

**$\beta$ -Naphthafuranone-2** (2-Hydroxy-1-naphthylacetic lactone, 4 : 5-benzisocoumaranone)



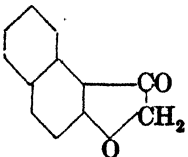
C<sub>12</sub>H<sub>8</sub>O<sub>2</sub> MW, 184

Leaflets. M.p. 107° (104°). Very spar. sol. H<sub>2</sub>O. Sol. conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  green fluor.

I.G., D.R.P., 562,391; E.P., 330,916, (*Chem. Abstracts*, 1933, **27**, 735; 1930, **24**, 6031).

Mayer, Schäfer, Rosenbach, *Chem. Zentr.*, 1929, II, 3009.

**$\beta$ -Naphthafuranone-3** (4 : 5-Benzcoumaranone)



C<sub>12</sub>H<sub>8</sub>O<sub>2</sub> MW, 184

Colourless needles from pet. ether, EtOH, or

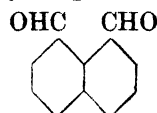
AcOH. M.p. 133°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with pale yellow col. Sol. alc. NaOH with bluish-red col. HNO<sub>3</sub>  $\rightarrow$  2-nitroderiv., m.p. 190° decomp.

2-p-Nitrobenzylidene deriv. : m.p. 270°.

Dziewoński, Duzyk, *Chem. Abstracts*, 1934, **28**, 4415.

Fries, Frelstedt, *Ber.*, 1921, **54**, 715.

**Naphthalaldehyde** (Naphthalene 1 : 8-dialdehyde, 1 : 8-dialdehydonaphthalene)



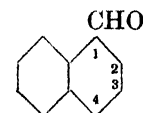
C<sub>12</sub>H<sub>8</sub>O<sub>2</sub> MW, 184

Hydrate : C<sub>12</sub>H<sub>8</sub>O<sub>2</sub>·H<sub>2</sub>O. Colourless needles from C<sub>6</sub>H<sub>6</sub>. M.p. 130°. Reduces NH<sub>3</sub>·AgNO<sub>3</sub>.

Di-p-nitrophenylhydrazone : reddish needles. M.p. 229°.

Criegee, Kraft, Rank, *Ann.*, 1933, **507**, 194.

**1-Naphthaldehyde** (Naphthalene  $\alpha$ -aldehyde)



C<sub>11</sub>H<sub>8</sub>O MW, 156

M.p. 33-4°. B.p. 292°, 156°/19 mm., 150°/9 mm. Forms bisulphite comp. Ox. in air  $\rightarrow$  1-naphthoic acid.

Anil : m.p. 71°.

Azine : m.p. 152°.

Oxime : m.p. 98° (39°).

Phenylhydrazone : m.p. 80°.

p-Nitrophenylhydrazone : m.p. 234°.

Semicarbazone : m.p. 221°.

Picrate : m.p. 94°.

Shoppee, *J. Chem. Soc.*, 1933, 42.

Wuyts et al., *Bull. soc. chim. Belg.*, 1932, **41**, 196; 1931, **40**, 665.

I.G., E.P., 250,955, (*Chem. Abstracts*, 1927, **21**, 1272).

Stephen, *J. Chem. Soc.*, 1925, 127, 1877.

Weil, Ostermeier, *Ber.*, 1921, **54**, 3217.

Gattermann, *Ann.*, 1912, **393**, 227.

**2-Naphthaldehyde** (Naphthalene  $\beta$ -aldehyde).

Leaflets from boiling H<sub>2</sub>O. M.p. 61° (59°). Sol. EtOH, Et<sub>2</sub>O. Volatile in steam. Gives bisulphite comp. KMnO<sub>4</sub>  $\rightarrow$  2-naphthoic acid.

Anil : m.p. 113°.

Azine : m.p. 232°.

Phenylhydrazone : m.p. 217-18° (205-6°).

## Naphthaldehyde-carboxylic Acid

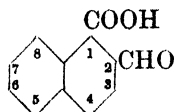
p-Nitrophenylhydrazone : m.p. 230°.  
Semicarbazone : m.p. 245°.

See first two and last two references above.

### Naphthaldehyde-carboxylic Acid.

See Naphthaldehydic Acid.

**1 : 2-Naphthaldehydic Acid** (2-Formyl-1-naphthoic acid, naphthalene-2-aldehyde-1-carboxylic acid, 2-aldehyde-1-naphthoic acid, 2-naphthaldehyde-1-carboxylic acid)



$C_{12}H_8O_3$

MW, 200

M.p. 176°.

Oxime : m.p. 215°.

Mayer, Schäfer, Rosenbach, *Chem. Abstracts*, 1930, **24**, 839.

**1 : 8-Naphthaldehydic Acid** (8-Formyl-1-naphthoic acid, naphthalene-8-aldehyde-1-carboxylic acid, 8-aldehyde-1-naphthoic acid, 1-naphthaldehyde-8-carboxylic acid).

Leaflets from EtOH.Aq. M.p. 167-8° decomp. (rapid heat.).

Me ester :  $C_{13}H_{10}O_3$ . MW, 214. Rhombic cryst. M.p. 105°. Mod. sol. hot  $H_2O$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

Acetyl deriv. of lactone form :  $C_{14}H_{10}O_4$ . MW, 242. M.p. 140°.

Graebe, Gfeller, *Ann.*, 1893, **276**, 13.

Zink, *Monatsh.*, 1901, **22**, 988.

Winterstein, Maxim, *Helv. Chim. Acta*, 1919, **2**, 202.

## Naphthalene



$C_{10}H_8$

MW, 128

Colourless plates from EtOH. M.p. 80-3°. B.p. 218°. Sublimes at 50°/760 mm., 22°/7 mm. Volatile in steam.  $D^{20}_D$  1.1517,  $D^{20}_L$  0.9625.  $n^{20}_D$  1.58232. Heat of comb.  $C_p$  and  $C_v$  1242 Cal. (9605 cal./gm.). Very sol.  $Et_2O$ ,  $C_6H_6$ , toluene, xylene, chlorobenzene, tetralin, hot EtOH,  $CHCl_3$ . Mod. sol. MeOH, cold EtOH. Spar. sol. cold pet. ether. Insol.  $H_2O$ . Forms add. comps. with 1 : 3-dinitrobenzene, m.p. 52°; 2 : 4-dinitrophenol, m.p. 95°; 2 : 4-dinitrotoluene, m.p. 61°; 2 : 4 : 6-trinitrotoluene, m.p. 97°. Passed through red-hot tube  $\rightarrow$  2:2'-dinaphthyl.  $H_2SO_4 + HgSO_4$ , or air + (vanadium comps.)  $\rightarrow$  phthalic acid.  $KMnO_4$ .Aq.  $\rightarrow$  phthalonic

## Naphthalene-1 : 2-dicarboxylic Acid

acid.  $HNO_3$  or  $CrO_3$ .Aq.  $\rightarrow$  phthalic acid.  
 $CrO_3 + AcOH \rightarrow$  1 : 4-naphthoquinone + phthalic acid.

Picrate : m.p. 149-5°.

Styphnate : m.p. 168-9°.

Ward, *J. Phys. Chem.*, 1934, **38**, 761.

Hill, U.S.P., 1,819,680, (*Chem. Abstracts*, 1931, **25**, 5759).

Schroeter, U.S.P., 1,763,410, (*Chem. Abstracts*, 1930, **24**, 3803).

Salont, *Dyer and Calico Printer*, 1928, **60**, 208 (Review).

Weissenberger, *Z. angew. Chem.*, 1927, **40**, 776.

Vesely, Jakes, *Bull. soc. chim.*, 1923, **33**, 955.

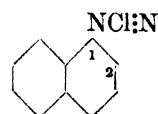
Davy, *Chem. Abstracts*, 1920, **14**, 618.

Bamberger, *Ber.*, 1913, **46**, 1899 (*Bibl.*).

### Naphthalene 1 : 8-Dialdehyde.

See Naphthalaldehyde.

**Naphthalene-1-diazonium chloride** ( $\alpha$ -Diazonaphthalene chloride)



$C_{10}H_7N_2Cl$

MW, 190.5

Pale yellow needles. M.p. 96° decomp. Sol.  $H_2O$ , AcOH, MeOH. Spar. sol. EtOH,  $Me_2CO$ . Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ , ligroin. Forms stable comp. with  $ZnCl_2$ .

Baudisch, Fürst, *Ber.*, 1912, **45**, 3428.

Badische, E.P., 238,676, (*Chem. Abstracts*, 1926, **20**, 1996).

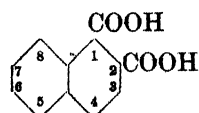
**Naphthalene-2-diazonium chloride** ( $\beta$ -Diazonaphthalene chloride).

Yellow needles. Explodes on heating. Sol.  $H_2O$ . Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ . Forms stable comp. with  $ZnCl_2$ .

Knoevenagel, *Ber.*, 1895, **28**, 2052, 2057.

See also second reference above.

### Naphthalene-1 : 2-dicarboxylic Acid



$C_{12}H_8O_4$

MW, 216

Cryst. from  $H_2O$ . M.p. 175°  $\rightarrow$  anhydride. Sol. EtOH,  $Et_2O$ , AcOH. Spar. sol.  $C_6H_6$ , ligroin,  $CHCl_3$ ,  $CS_2$ .

Di-Me ester :  $C_{14}H_{12}O_4$ . MW, 244. Cryst. from MeOH. M.p. 80°. Spar. sol. pet. ether.

**Diamide**:  $C_{12}H_{10}O_2N_2$ . MW, 214. Plates. M.p.  $265^\circ \rightarrow$  imide.

**Dinitrile**: 1 : 2-dicyanonaphthalene.  $C_{12}H_6N_4$ . MW, 178. Needles from  $C_6H_6$ . M.p.  $190^\circ$ . Sublimes. Spar. sol. EtOH.

**Anhydride**:  $C_{12}H_6O_3$ . MW, 198. Needles from EtOH. M.p.  $168-9^\circ$ . Sublimes. Sol.  $Et_2O$ .

**Imide**:  $C_{12}H_7O_2N$ . MW, 197. M.p.  $224^\circ$ .

Waldmann, Weiss, *J. prakt. Chem.*, 1930, **127**, 195.

Noto, *Gazz. chim. ital.*, 1915, **45**, ii, 126, 427.

Freund, Fleischer, *Ann.*, 1913, **399**, 212.

#### Naphthalene-1 : 4-dicarboxylic Acid.

Rodlets from AcOH. M.p.  $309^\circ$  ( $288^\circ$ ). Sol. EtOH  $\rightarrow$  blue fluor. Insol. boiling  $H_2O$ .

**Di-Me ester**: cryst. from AcOH. M.p.  $64^\circ$ . B.p.  $195-7^\circ/12$  mm.

**Dichloride**:  $C_{12}H_6O_2Cl_2$ . MW, 253. Needles from pet. ether. M.p.  $80^\circ$ .

**Dinitrile**: 1 : 4-dicyanonaphthalene. Needles from AcOH. M.p.  $206^\circ$ . Spar. sol. EtOH,  $Et_2O$ .

I.G., D.R.P., 558,471, (*Chem. Abstracts*, 1933, **27**, 310).

Mayer *et al.*, *Ber.*, 1922, **55**, 1841.

Scholl, Neumann, *ibid.*, 120.

#### Naphthalene-1 : 5-dicarboxylic Acid.

Colourless needles from  $PhNO_2$ . M.p.  $315-20^\circ$  decomp. Insol. ord. solvents.

**Di-Me ester**: leaflets from MeOH. M.p.  $114-15^\circ$ .

**Di-Et ester**:  $C_{16}H_{16}O_4$ . MW, 272. Needles. M.p.  $123-4^\circ$ .

**Diphenyl ester**:  $C_{24}H_{16}O_4$ . MW, 368. Cryst. from  $C_6H_6$ . M.p.  $198-9^\circ$ .

**Dichloride**: needles from  $CHCl_3$ . M.p.  $155-6^\circ$ .

**Dinitrile**: 1 : 5-dicyanonaphthalene. Needles from EtOH. M.p.  $260^\circ$  ( $267^\circ$ ). Sublimes.

Salkind, *Ber.*, 1934, **67**, 1031.

See also third reference above.

#### Naphthalene-1 : 6-dicarboxylic Acid.

Needles from AcOH. M.p.  $310^\circ$ . Sol. hot EtOH, hot AcOH.

**Di-Me ester**: needles from EtOH. M.p.  $99^\circ$ .

**Dinitrile**: 1 : 6-dicyanonaphthalene. Needles from EtOH. M.p.  $208-10^\circ$ .

Weissgerber, Kruber, *Ber.*, 1919, **52**, 354.

#### Naphthalene-1 : 7-dicarboxylic Acid.

Micro-cryst. powder from EtOH.Aq. M.p.  $294-6^\circ$  decomp. Sol. ord. org. solvents.

**Di-Me ester**: cryst. from EtOH. M.p.  $86-7^\circ$ .

Ruzicka, Melsen, *Helv. Chim. Acta*, 1931, **14**, 397.

#### Naphthalene-1 : 8-dicarboxylic Acid.

See Naphthalic Acid.

#### Naphthalene-2 : 3-dicarboxylic Acid.

Prisms from AcOH. M.p.  $239-41^\circ$  ( $246^\circ$  after sublimation). Sol. hot EtOH. Spar. sol.  $Et_2O$ , hot AcOH. Very spar. sol. ligroin,  $CHCl_3$ ,  $C_6H_6$ ,  $CS_2$ , cold  $H_2O$ . Heat + aniline  $\rightarrow$  phenylimide.  $SOCl_2$  or  $PCl_5 \rightarrow$  anhydride. The  $NH_4$  salt at  $270^\circ \rightarrow$  imide.

**Anhydride**: rectangular plates from AcOH. M.p.  $246^\circ$ .

**Mono-nitrile**: 3-cyano-2-naphthoic acid.  $C_{12}H_7O_2N$ . MW, 197. Yellow cryst. M.p.  $273-4^\circ$ .

**Imide**: micro-needles from  $CHCl_3$ -EtOH. M.p.  $275^\circ$  (softens at  $250^\circ$ ).

**Phenylimide**:  $C_{18}H_{11}O_2N$ . MW, 273. Rectangular silvery plates from  $CHCl_3$ -EtOH. M.p.  $277-8^\circ$ .

Waldmann, Mathiowetz, *Ber.*, 1931, **64**, 1713.

I.G., F.P., 682,474, (*Chem. Abstracts*, 1930, **24**, 4306).

Freund, Fleischer, *Ann.*, 1913, **402**, 68.

#### Naphthalene-2 : 6-dicarboxylic Acid.

Needles from EtOH.Aq. M.p. above  $300^\circ$  decomp. Insol. boiling  $C_6H_6$ , toluene, AcOH.

**Di-Me ester**: cryst. from MeOH. M.p.  $191^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ , toluene, hot ligroin. Spar. sol. pet. ether.

**Mono-nitrile**: 6-cyano-2-naphthoic acid. M.p. above  $300^\circ$  decomp. Very spar. sol. org. solvents.

**Dinitrile**: 2 : 6-dicyanonaphthalene. Needles from AcOH. M.p.  $296-7^\circ$ . Insol. boiling EtOH,  $Et_2O$ ,  $C_6H_6$ .

**Dianilide**: leaflets from aniline. Does not melt below  $320^\circ$ .

Kaufler, Thien, *Ber.*, 1907, **40**, 3257.

#### Naphthalene-2 : 7-dicarboxylic Acid.

Needles from EtOH. M.p. above  $300^\circ$  decomp. Very spar. sol. boiling  $C_6H_6$ , toluene, AcOH.

**Di-Me ester**: cryst. from MeOH. M.p.  $135-6^\circ$  ( $141^\circ$ ). Sol. hot ligroin. Spar. sol. pet. ether.

**Di-Et ester**: m.p.  $238^\circ$ .

**Diphenyl ester**:  $C_{24}H_{16}O_4$ . MW, 368. M.p.  $162^\circ$ .

**Mono-nitrile**: 7-cyano-2-naphthoic acid. M.p. above  $300^\circ$  decomp.

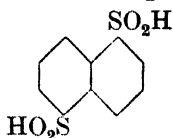
**Dinitrile**: 2 : 7-dicyanonaphthalene. Needles from AcOH. M.p.  $267-8^\circ$ . Sol. hot EtOH.

**Dianilide**: scales from aniline. M.p.  $297-8^\circ$ .

Purgotti, *Chem. Abstracts*, 1926, **20**, 1618. See also previous reference.

**Naphthalene-dihydride.**

See Dihydronaphthalene.

**Naphthalene-1 : 5-disulphinic Acid** $C_{10}H_8O_4S_2$ 

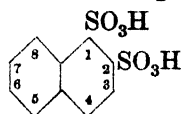
MW, 256

Glittering leaflets from HCl.Aq. M.p. 166–7° (174–5° decomp.). Sol. hot  $H_2O$ , hot EtOH. Spar. sol.  $Et_2O$ , cold EtOH, cold  $H_2O$ . Reduces cold alk.  $KMnO_4$ . Reacts with *p*-benzoquinone (2 mols.)  $\longrightarrow$  comp., m.p. 294°.

*Di-NH<sub>4</sub> salt*: prisms from EtOH. M.p. 194°.

Curtius, Tüxen, *J. prakt. Chem.*, 1930, 125, 406.

Corbellini, Albenga, *Gazz. chim. ital.*, 1931, 61, 111.

**Naphthalene-1 : 2-disulphonic Acid** $C_{10}H_8O_6S_2$ 

MW, 288

*Anhydride*:  $C_{10}H_6O_5S_2$ . MW, 270. M.p. 198–9°.

Gattermann, *Ber.*, 1899, 32, 1156.

**Naphthalene-1 : 3-disulphonic Acid.**

*Dichloride*:  $C_{10}H_6O_4Cl_2S_2$ . MW, 325. Prisms from  $C_6H_6$ . M.p. 138°.

Dressel, Kothe, *Ber.*, 1894, 27, 1197.

Armstrong, Wynne, *Chem. News*, 1890, 62, 163.

**Naphthalene-1 : 4-disulphonic Acid.**

*Dichloride*: plates. M.p. 160°. Sol.  $C_6H_6$ .

*Diamide*:  $C_{10}H_{10}O_4N_2S_2$ . MW, 286. Needles from EtOH.Aq. M.p. 273°.

*Dianilide*: pearly leaflets. M.p. 179°.

Gattermann, *Ber.*, 1899, 32, 1156.

**Naphthalene-1 : 5-disulphonic Acid ("γ"-Naphthalenedisulphonic acid).**

Plates +  $4H_2O$  from HCl.Aq. M.p. anhyd. 240–5°. Sol. 0.98 part  $H_2O$  at 20°. Aq. sol. tastes bitter-astringent. Forms series of arylamine salts of definite m.ps.

*Di-Me ester*:  $C_{12}H_{12}O_6S_2$ . MW, 316. Cryst. from  $CHCl_3$ . M.p. 205°.

*Mono-Et ester*:  $C_{12}H_{12}O_6S_2$ . MW, 316. Cryst. from EtOH. M.p. 147°.

*Difluoride*:  $C_{10}H_6O_4F_2S_2$ . MW, 292. M.p. 203°.

*Dichloride*: prisms from  $C_6H_6$ . M.p. 183°.

*Diamide*: does not melt below 340°.

*Dianilide*: m.p. 248–9°.

*Diazide*:  $C_{10}H_6O_4N_6S_2$ . MW, 338. Cryst. from AcOH or  $CHCl_3$ . M.p. 177°.

*Dihydrazide*:  $C_{10}H_{12}O_4N_4S_2$ . MW, 316. Micro-needles from  $H_2O$ . Does not melt (blackens at 240°).

*Benzyl-ψ-thiourea salt*: m.p. 251° decomp.

Corbellini, Albenga, *Gazz. chim. ital.*, 1931, 61, 111.

Curtius, Tüxen, *J. prakt. Chem.*, 1930, 125, 401.

Steinkopf *et al.*, *ibid.*, 1927, 117, 1.

Lynch, Scanlan, *Ind. Eng. Chem.*, 1927, 19, 1010.

Fierz-David, Hasler, *Helv. Chim. Acta*, 1923, 6, 1133.

Forster, Hishiyama, *J. Soc. Chem. Ind.*, 1932, 51, 297r.

Hann, Keenan, *J. Phys. Chem.*, 1927, 31, 1086.

**Naphthalene-1 : 6-disulphonic Acid ("δ"-Naphthalenedisulphonic acid).**

Prisms +  $4H_2O$  from  $H_2O$ . M.p. anhyd. 125° decomp. Sol. 0.61 part  $H_2O$  at 18–20°. Forms α-naphthylamine salt, m.p. 265–7° decomp.

*Dichloride*: leaflets from  $C_6H_6$ . M.p. 129°. Very sol.  $C_6H_6$ .

*Diamide*: m.p. 297–8°.

*Benzyl-ψ-thiourea salt*: decomp. at 81°.

Ufimzew, Kriwoschlukowa, *J. prakt. Chem.*, 1934, 140, 172.

Ambler, *Ind. Eng. Chem.*, 1927, 19, 417; 1920, 12, 1080.

See also last three references above.

**Naphthalene-1 : 7-disulphonic Acid.**

*Dichloride*: prisms from  $C_6H_6$ . M.p. 123°. Sol. 7% in  $C_6H_6$ .

See first reference above.

**Naphthalene-1 : 8-disulphonic Acid.**

*Anhydride*: plates. M.p. 227°. Sol. hot AcOH, hot xylene. Spar. sol.  $C_6H_6$ .

Armstrong, Wynne, *Chem. News*, 1893, 67, 299.

**Naphthalene-2 : 6-disulphonic Acid ("β"-Naphthalenedisulphonic acid).**

Leaflets. Deliquesces very slowly in air. Forms series of arylamine salts of definite m.ps.

*Dichloride*: flat needles. M.p. 225°. Spar. sol.  $C_6H_6$ . Insol.  $Et_2O$ .

*Benzyl-ψ-thiourea salt* : m.p. 256°.

Hann, Keenan, *J. Phys. Chem.*, 1927, **31**, 1086.

Heid, *J. Am. Chem. Soc.*, 1927, **49**, 844.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1924 **43**, 165r.

Fierz-David, Hasler, *Helv. Chim. Acta*, 1923, **6**, 1133.

Armstrong, Wynne, *Chem. News*, 1890, **62**, 163.

**Naphthalene-2 : 7-disulphonic Acid** ("α"-*Naphthalenedisulphonic acid*).

Very hygroscopic needles. Spar. sol. cold conc. HCl. Forms series of arylamine salts of definite m.ps.

*Dichloride* : four-sided plates from C<sub>6</sub>H<sub>6</sub>. M.p. 159°. Mod. sol. Et<sub>2</sub>O.

*Dibromide* : C<sub>10</sub>H<sub>6</sub>O<sub>4</sub>Br<sub>2</sub>S<sub>2</sub>. MW, 414. Prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 137°.

*Diamide* : needles. M.p. 242-3°.

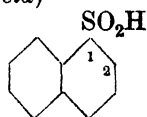
*Benzyl-ψ-thiourea salt* : m.p. 211-12° decomp.

Ufimzew, Kriwoschlukowa, *J. prakt. Chem.*, 1934, **140**, 172.

Ambler, *Ind. Eng. Chem.*, 1920, **12**, 1194.

See also first four references above.

**Naphthalene-1-sulphinic Acid** (α-*Naphthalenesulphinic acid*)



C<sub>10</sub>H<sub>8</sub>O<sub>2</sub>S MW, 192

Needles from H<sub>2</sub>O. M.p. 98-9° (84-5°). Sol. H<sub>2</sub>O. Mod. sol. EtOH. Spar. sol. HCl.Aq., Et<sub>2</sub>O. Dil. HCl at 180° → naphthalene + SO<sub>2</sub>.

Höchst, D.R.P., 224,019, (*Chem. Zentr.*, 1910, II, 513).

Knoevenagel, Kenner, *Ber.*, 1908, **41**, 3319.

Rosenheim, Singer, *Ber.*, 1904, **37**, 2154.

Otto, Rössing, Troeger, *J. prakt. Chem.*, 1893, **47**, 95.

Thomas, *J. Chem. Soc.*, 1909, **95**, 342.

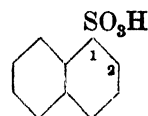
**Naphthalene-2-sulphinic Acid** (β-*Naphthalenesulphinic acid*).

Needles from H<sub>2</sub>O. M.p. 105°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Sol. in conc. H<sub>2</sub>SO<sub>4</sub> gradually turns green. Dil. HCl at 150° → naphthalene + SO<sub>2</sub>.

*Me ester* : C<sub>11</sub>H<sub>10</sub>O<sub>2</sub>S. MW, 206. Leaflets from pet. ether. M.p. 44°. Sol. ord. org. solvents. Decomp. on standing. Hyd. by H<sub>2</sub>O.

See last two references above.

**Naphthalene-1-sulphonic Acid** (*Naphthalene-α-sulphonic acid*)



C<sub>10</sub>H<sub>8</sub>O<sub>3</sub>S MW, 208

Prisms + 2H<sub>2</sub>O from HCl.Aq. M.p. 90°. Sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O.  $k = 0.18 \times 10^{-3}$  at 25°. Forms series of arylamine salts of definite m.ps. Acid KMnO<sub>4</sub> → phthalic acid.

*Me ester* : C<sub>11</sub>H<sub>10</sub>O<sub>3</sub>S. MW, 222. M.p. 78° (72-3°). B.p. 214°/15 mm.

*Et ester* : C<sub>12</sub>H<sub>12</sub>O<sub>3</sub>S. MW, 236. Liq. Decomp. on dist.

*Phenyl ester* : C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>S. MW, 284. M.p. 75°.

*Fluoride* : C<sub>10</sub>H<sub>7</sub>O<sub>2</sub>FS. MW, 210. M.p. 56°.

*Chloride* : C<sub>10</sub>H<sub>7</sub>O<sub>2</sub>ClS. MW, 226.5. Leaflets from Et<sub>2</sub>O. M.p. 68°. B.p. 194-5°/13 mm., 147.5°/0.9 mm.

*Bromide* : C<sub>10</sub>H<sub>7</sub>O<sub>2</sub>BrS. MW, 271. M.p. 88-9°.

*Amide* : C<sub>10</sub>H<sub>9</sub>O<sub>2</sub>NS. MW, 207. M.p. 150°.

*Anilide* : m.p. 152°.

*Azide* : cryst. from EtOH. M.p. 53°, decomp. at 133°.

*Hydrazide* : needles from EtOH.Aq. M.p. 123° decomp. *HCl salt* : m.p. 142°.

*Piperidide* : m.p. 133-4°.

*Benzyl-ψ-thiourea salt* : m.p. 138°.

*2-Naphthylamine salt* : m.p. 276-9° decomp.

Geigy F.P., 765,771, (*Chem. Abstracts*, 1934, **28**, 6726).

Cumming, Muir, *Chem. Abstracts*, 1934, **28**, 4409.

Masters, U.S.P., 1,922,813, (*Chem. Abstracts*, 1933, **27**, 5085).

Radcliffe, Short, *J. Chem. Soc.* 1931, 220.

Curtius, Bottler, Hasse, *J. prakt. Chem.*, 1930, **125**, 366.

Fierz-David, Weissenbach, *Helv. Chim. Acta*, 1920, **3**, 310, 315.

Rodionow, *Bull. soc. chim.*, 1929, **45**, 117.

Hann, Keenan, *J. Phys. Chem.*, 1927, **31**, 1084.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1924, **43**, 299r.

**Naphthalene-2-sulphonic Acid** (*Naphthalene-β-sulphonic acid*).

Very hygroscopic cryst. M.p. 91°. On standing in air, or cryst. from HCl.Aq. → trihydrate, m.p. 83°. Kept over CaCl<sub>2</sub> or conc. H<sub>2</sub>SO<sub>4</sub> → monohydrate, m.p. 124°.  $k = 0.25 \times 10^{-3}$  at 25°. Very sol. ord. org. solvents. Forms series of arylamine salts of definite m.ps.



## Naphthalene-1 : 4 : 5 : 8-tetracarboxylic Acid

With 1 mol. glycine  $\rightarrow$  cryst. comp., m.p. 193°. Neutral or acid  $\text{KMnO}_4 \rightarrow$  phthalic acid.

*Me ester* : m.p. 56°. B.p. 224-5°/15 mm.

*Et ester* : m.p. 11-12°. B.p. 134°/vac. of cathode light.

*Phenyl ester* : m.p. 98-9°.

*Fluoride* : m.p. 87-8°.

*Chloride* : m.p. 79° (66°). B.p. 201°/13 mm., 148°/0.6 mm.

*Bromide* : m.p. 96-7°.

*Amide* : m.p. 217° (212°).

*Azide* : needles from ligroin. M.p. 44-6° slight decomp.

*Hydrazide* : m.p. 137-9°. *HCl salt* : m.p. 148-50°.

*Disulphonimide* :  $(\text{C}_{10}\text{H}_7\text{SO}_2)_2\text{NH}$ . (Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 180-1°. Sol.  $\text{H}_2\text{O}$ .)

*Benzyl- $\psi$ -thiourea salt* : m.p. 193°.

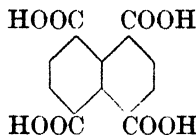
*2-Naphthylamine salt* : m.p. 211° (brown at 202°).

I.G., D.R.P., 574,836; E.P., 384,722, (*Chem. Abstracts*, 1933, 27, 4543, 4251).

Dennis, U.S.P., 1,332,203, (*Chem. Abstracts*, 1920, 14, 1123).

See also last three references above.

## Naphthalene-1 : 4 : 5 : 8-tetracarboxylic Acid



$\text{C}_{14}\text{H}_8\text{O}_8$

MW, 304

Leaflets or needles from  $\text{HCl.Aq.}$  No characteristic m.p. Rapid heat at 200-50°  $\rightarrow$  decomp., slow heat at 140-50° or cryst. from  $\text{AcOH} \rightarrow$  anhydride. Sol.  $\text{Me}_2\text{CO.Aq.}$  Mod. sol.  $\text{H}_2\text{O}$ , hot  $\text{AcOH}$ . Very spar. sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{EtOH}$ .

*Di-anhydride* :  $\text{C}_{14}\text{H}_4\text{O}_6$ . MW, 266. Needles from  $\text{AcOH}$ . Sublimes above 300°. Heat +  $\text{NH}_3 \rightarrow$  di-imide.

*Di-imide* :  $\text{C}_{14}\text{H}_6\text{O}_4\text{N}_2$ . MW, 266. Yellowish needles from  $\text{H}_2\text{O}$ . Sublimes above 270°. Very spar. sol. ord. org. solvents.

Greune, Eckert, U.S.P., 1,970,651, (*Chem. Abstracts*, 1934, 28, 6159).

I.G., D.R.P., 601,104; F.P., 756,156, (*Chem. Abstracts*, 1934, 28, 7267, 2018).

F.P., 721,339, (*Chem. Abstracts*, 1932,

26, 4184). E.P., 364,116, (*Chem. Abstracts*, 1933, 27, 2457).

E.P., 363,044, (*Chem. Abstracts*, 1933, 27, 1642).

Freund, Fleischer, *Ann.*, 1913, 402, 74.

Bamberger, Philip, *Ann.*, 1887, 240, 182.

## Naphthalene-1 : 3 : 5-trisulphonic Acid

## Naphthalene-1 : 3 : 5 : 7-tetrasulphonic Acid



$\text{C}_{10}\text{H}_8\text{O}_{12}\text{S}_4$

MW, 448

*Ba salt* :  $\text{C}_{10}\text{H}_4(\text{SO}_3)_4\text{Ba}_2$ . Dimorphous. Blunt prisms +  $14\text{H}_2\text{O}$  at 15° (efflorescent). Cryst. +  $8\text{H}_2\text{O}$  at 35° (stable in air).

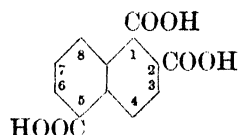
*Tetrachloride* :  $\text{C}_{10}\text{H}_4\text{O}_8\text{Cl}_4\text{S}_4$ . MW, 522. Tetrahedral cryst. M.p. 261-2°. Spar. sol.  $\text{C}_6\text{H}_6$ ,  $\text{Me}_2\text{CO}$ .

Schmid, *Chem. Abstracts*, 1922, 16, 2141.

Fierz David, *J. Soc. Chem. Ind.*, 1923, 42, 421T.

Cf. Ufimzew, Kriwoschlükowa, *J. prakt. Chem.*, 1934, 140, 172.

## Naphthalene-1 : 2 : 5-tricarboxylic Acid



$\text{C}_{13}\text{H}_8\text{O}_6$

MW, 260

Colourless feathery needles. M.p. (vac. sublimed) 270-2°. Sol.  $\text{MeOH}$ .

*Tri-Me ester* :  $\text{C}_{16}\text{H}_{14}\text{O}_6$ . MW, 302. Cryst. M.p. 91-2°.

Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 2546.

Ruzicka, Hosking, *Helv. Chim. Acta*, 1930, 13, 1405, 1411.

## Naphthalene-1 : 4 : 5-tricarboxylic Acid.

Cryst. from conc.  $\text{HCl}$ . Does not melt, but at 100-20° forms the anhydride.

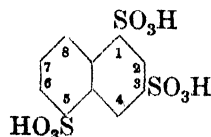
*Anhydride* :  $\text{C}_{13}\text{H}_6\text{O}_5$ . MW, 242. Cryst. from  $\text{EtOH.Aq.}$  M.p. 274° (243°).

*Anhydride Me ester* :  $\text{C}_{14}\text{H}_8\text{O}_5$ . MW, 256. Needles from  $\text{AcOH}$ . M.p. 222°.

Fieser Peters, *J. Am. Chem. Soc.*, 1932, 54, 4352.

Graebe, Haas, *Ann.*, 1903, 327, 95.

## Naphthalene-1 : 3 : 5-trisulphonic Acid



$\text{C}_{10}\text{H}_8\text{O}_9\text{S}_3$

MW, 368

Amorph. mass. Readily takes up  $\text{H}_2\text{O} \rightarrow$

oily liq. which carbonises cellulose and decomposes NaCl.

*Trichloride*:  $C_{10}H_5O_6Cl_3S_3$ . MW, 423.5. Cryst. from  $C_6H_6$ -ligroin. M.p. 146°.

Erdmann, *Ber.*, 1899, **32**, 3188.

Gattermann, *ibid.*, 1158.

### Naphthalene-1 : 3 : 6-trisulphonic Acid.

*Pb salt*: very sol.  $H_2O$ .

*Trichloride*: prisms from  $(C_6H_6)$ -pet. ether. M.p. 194°.

Armstrong, Wynne, *Chem. News*, 1888, **57**, 9; 1890, **62**, 162.

Cf. Ufimzew, Kriwoschlikowa, *J. prakt. Chem.*, 1934, **140**, 172.

See also first reference above.

### Naphthalene-1 : 4 : 5-trisulphonic Acid.

*Na salt*: cryst. Effloresces in air. Very sol.  $H_2O$ .

*Trichloride*: needles. M.p. 156-7°.

Gattermann, *Ber.*, 1899, **32**, 1139, 1158.

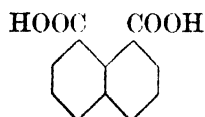
### Naphthalene-2 : 3 : 6-trisulphonic Acid.

*Na salt*: spar. sol.  $H_2O$ .

*Trichloride*: plates from  $C_6H_6$ . M.p. 200°.

Armstrong, Wynne, *Chem. News*, 1893, **67**, 299.

**Naphthalic Acid** (*Naphthalene-1 : 8-dicarboxylic acid*)



$C_{12}H_8O_4$  MW, 216

Needles from EtOH. Sol. warm EtOH. Spar. sol.  $Et_2O$ . Insol.  $H_2O$ .

*Di-Me ester*:  $C_{14}H_{12}O_4$ . MW, 244. Prisms from MeOH.Aq. M.p. 102-3°.

*Di-Et ester*:  $C_{16}H_{16}O_4$ . MW, 272. Leaflets from EtOH.Aq. M.p. 59-60°. B.p. 238-9°/19 mm. Sol. in conc.  $H_2SO_4$   $\rightarrow$  blue fluor.

*Dibutyl ester*:  $C_{20}H_{24}O_4$ . MW, 328. Cryst. from MeOH.Aq. M.p. 52-3°.

*Dichloride*: naphthalyl chloride.  $C_{12}H_6O_2Cl_2$ . MW, 253. Prisms from  $CS_2$ . M.p. 84-6°. B.p. 195-200°/0.2 mm.

*Mono-nitrile*: 8-cyano-1-naphthoic acid.  $C_{12}H_7O_2N$ . MW, 197. M.p. 210-50° decomp.

*Anhydride*:  $C_{12}H_6O_3$ . MW, 198. Needles from EtOH. M.p. 274°.

*Imide*:  $C_{12}H_7O_2N$ . MW, 197. M.p. 300° (290-1°).

*N-Me imide*:  $C_{13}H_9O_2N$ . MW, 211. M.p. 205°.

*N-Et imide*:  $C_{14}H_{11}O_2N$ . MW, 225. M.p. 148°.

*N-Phenyl imide*:  $C_{18}H_{11}O_2N$ . MW, 273. M.p. 202°.

*Dianilide*: m.p. 250-82° decomp.

*Di-Me anilide*: m.p. 245-6°.

Duckert, *Chem. Abstracts*, 1934, **28**, 1255 (*Bibl.*).

Jaeger, Canadian P., 321,683, (*Chem. Abstracts*, 1932, **26**, 3263).

Davies, Leeper, *J. Chem. Soc.*, 1927, 1124.

Mason, *J. Chem. Soc.*, 1924, **125**, 2116.

Wislicenus, Penndorf, *Ber.*, 1912, **45**, 410.

Graebe, Gfeller, *Ber.*, 1892, **25**, 653; *Ann.*, 1893, **276**, 6.

### Naphthamide.

See under Naphthoic Acid.

### Naphthane.

See Decahydronaphthalene.

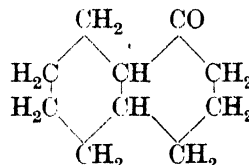
### $\beta$ -Naphthanene.

See 1 : 2 : 3 : 4 : 5 : 8 : 9 : 10-Octahydronaphthalene.

### Naphthanol.

See Decahydronaphthol.

$\alpha$ -Naphthanone (1-Ketodecahydronaphthalene)



$C_{10}H_{16}O$

MW, 152

Prismatic plates with pronounced menthol-like odour. M.p. 32°. Sol. ord. org. solvents. Spar. sol.  $H_2O$ . Gives unstable comp. with  $NaHSO_3$ .

*Oxime*: cryst. from EtOH. M.p. 165°. Sublimes in needles at 100°.

*Semicarbazone*: needles. M.p. about 230°.

Leroux, *Ann. chim. phys.*, 1910, **21**, 522.

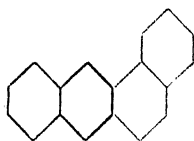
$\beta$ -Naphthanone (2-Ketodecahydronaphthalene).

Colourless liq. with strong unpleasant odour. B.p. 240°, 110°/15 mm.  $D_{16}^{20}$  0.979.  $n_D^{20}$  1.4834. Sol. EtOH,  $Et_2O$ , AcOH. Spar. sol.  $H_2O$ . Reduces Fehling's and  $NH_3.AgNO_3$ .

*Oxime*: prisms from pet. ether. M.p. 76°. Sol. EtOH,  $Et_2O$ . Spar. sol. pet. ether.

*Semicarbazone*: needles from EtOH. M.p. 195°.

See previous reference.

**Naphthanthracene** (1 : 2-Benzanthracene) $C_{18}H_{12}$ 

MW, 228

Leaflets from EtOH-AcOH. M.p. 158-9° (141°). Sublimes. Sols. fluor. intense yellowish-green.  $Na_2Cr_2O_7 + AcOH \rightarrow$  naphthanthraquinone.

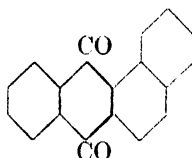
Picrate : red needles. M.p. 133°.

Barnett, Matthews, *Chem. News*, 1925, 130, 339.

Gabriel, Colman, *Ber.*, 1900, 33, 447.

Graebe, *Ann.*, 1905, 340, 258.

**Naphthanthraquinone** (ang-Benzanthraquinone, 1 : 2-benzanthraquinone, 1 : 2-phthaloylnaphthalene)

 $C_{18}H_{10}O_2$ 

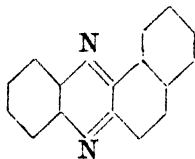
MW, 258

Yellow cryst. from hot xylene or  $PhNO_2$ . M.p. 168°. Sol.  $C_6H_6$ ,  $CHCl_3$ , toluene. Spar. sol. most other solvents. Conc.  $H_2SO_4 \rightarrow$  olive-green sol.  $KMnO_4 \rightarrow$  anthraquinone-1 : 2-dicarboxylic acid.  $Zn + NH_3 \rightarrow$  naphthanthracene.

Dziewoński, Ritt, *Chem. Abstracts*, 1928, 22, 2561.

See also second reference above.

**ang-Naphthaphenazine** ("  $\alpha$  "-Benzophenazine)

 $C_{16}H_{10}N_2$ 

MW, 230

Bright yellow needles. M.p. 142°. Dist. un-decomp. above 360°. Sol. EtOH,  $Et_2O$ , AcOH. Insol.  $H_2O$ . Sol. conc.  $H_2SO_4 \rightarrow$  reddish-brown; on dilution  $\rightarrow$  golden-yellow col. + cryst. ppt. of the azine.

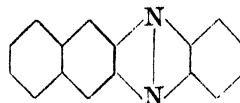
Ethiodide :  $C_{18}H_{15}N_2I$ . MW, 386. Black needles with violet reflex. M.p. 150° decomp.

N-Oxide :  $C_{18}H_{10}ON_2$ . MW, 246. Green cryst. M.p. 182°.

Kehrmann, Mermod, *Helv. Chim. Acta*, 1927, 10, 64.

Fischer, Hepp, *Ber.*, 1897, 30, 393.

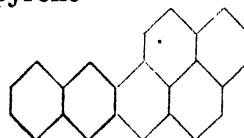
**lin-Naphthaphenazine** ("  $\beta$  "-Benzophenazine)

 $C_{16}H_{10}N_2$ 

MW, 230

Red leaflets from  $C_6H_6$ . M.p. 233° (darkens). Sol.  $CHCl_3$ ,  $C_6H_6$ , hot AcOH. Spar. sol. EtOH. Conc.  $H_2SO_4 \rightarrow$  yellow sol.

Hinsberg, *Ann.*, 1901, 319, 261.

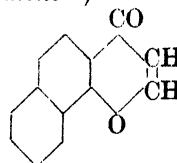
**Naphthapyrene** $C_{24}H_{14}$ 

MW, 302

Deep orange leaflets from  $C_6H_6$ . M.p. 273°.

Cook, Hewett, *J. Chem. Soc.*, 1933, 403.

**$\alpha$  :  $\beta$ -Naphthapyrone** (7 : 8-Benzchromone, " $\alpha$ -naphthochromone")

 $C_{13}H_8O_2$ 

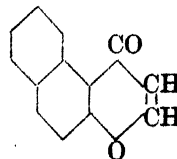
MW, 196

Needles from EtOH.Aq. M.p. 125°. Yellow sol. in conc.  $H_2SO_4 \rightarrow$  intense bluish-green fluor.

Semicarbazone : m.p. 256° decomp.

Pfeiffer, Grimmer, *Ber.*, 1917, 50, 922.

**$\beta$  :  $\alpha$ -Naphthapyrone** (5 : 6-Benzchromone, " $\beta$ -naphthochromone")

 $C_{13}H_8O_2$ 

MW, 196

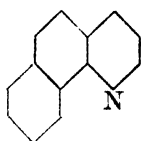
Stout pale yellow needles from pet. ether. M.p. 103°. Sol. EtOH,  $C_6H_6$ , AcOH. Spar.

## $\alpha$ -Naphthaquinoline

sol. cold pet. ether. The colourless sol. in boiling  $\text{AcOH} + 1$  drop conc.  $\text{H}_2\text{SO}_4 \rightarrow$  blue fluor.

Menon, Venkataraman, *J. Chem. Soc.*, 1931, 2593.

**$\alpha$ -Naphthaquinoline** (7 : 8-Benzquinoline, naphtha-1' : 2' : 2 : 3-pyridine)



$\text{C}_{13}\text{H}_9\text{N}$

MW, 179

Plates from pet. ether. M.p.  $52^\circ$ . B.p.  $338^\circ/719$  mm.,  $223^\circ/47$  mm. Sol. ord. org. solvents. Insol.  $\text{H}_2\text{O}$ . Volatile with superheated steam.

$\text{B}_2\text{HCl}$ : yellowish needles. M.p.  $213^\circ$ .

$\text{B}_2\text{H}_2\text{PtCl}_6$ : bright yellow prisms. M.p.  $224^\circ$ . Very spar. sol.  $\text{H}_2\text{O}$ .

Methiodide:  $\text{C}_{14}\text{H}_{12}\text{NI}$ . MW, 321. M.p.  $179^\circ$ .

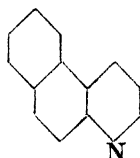
Picrate: m.p.  $191-2^\circ$ .

Stewart, *J. Chem. Soc.*, 1925, 127, 1332.

Haid, *Monatsh.*, 1906, 27, 318.

Claus, Imhoff, *J. prakt. Chem.*, 1898, 57, 68.

**$\beta$ -Naphthaquinoline** (5 : 6-Benzquinoline, naphtha-2' : 1' : 2 : 3-pyridine)



$\text{C}_{13}\text{H}_9\text{N}$

MW, 179

Leaflets from  $\text{H}_2\text{O}$  or pet. ether. M.p.  $94^\circ$ . B.p.  $350^\circ/721$  mm.,  $210-15^\circ/22$  mm. Difficultly volatile in steam. Sol. ord. org. solvents. Very spar. sol.  $\text{H}_2\text{O}$ . Sol. dil.  $\text{HCl}$  with fluor.

$\text{B}_2\text{H}_2\text{SO}_4$ : yellow needles. M.p.  $90^\circ$ .

Methiodide:  $\text{C}_{14}\text{H}_{12}\text{NI}$ ,  $2\text{H}_2\text{O}$ . M.p.  $200-5^\circ$  decomp. ( $186^\circ$ ).

Ethiodide:  $\text{C}_{15}\text{H}_{14}\text{NI}$ . MW, 335. M.p.  $206^\circ$  decomp.

Benzyl chloride quaternary salt:  $\text{C}_{20}\text{H}_{16}\text{NCl}$ ,  $2\text{H}_2\text{O}$ . M.p. anhyd.  $196^\circ$ .

Picrate: m.p.  $259^\circ$  ( $251-2^\circ$ ).

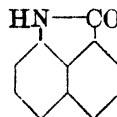
Bamberger, Müller, *Ber.*, 1891, 24, 2643.

Knueppel, *Ber.*, 1896, 29, 708.

Braun, Gruber, *Ber.*, 1922, 55, 1714.

## 11 1 : 8-Naphthasultam-4-sulphonic Acid

**Naphthastyryl** (8-Amino-1-naphtholactam, peri-naphthazolone)



$\text{C}_{11}\text{H}_7\text{ON}$

MW, 169

Fine needles from  $\text{EtOH}$ . M.p.  $180-1^\circ$ . Sublimes in yellow needles. Sol.  $\text{EtOH} \rightarrow$  green fluor. Mod. sol. boiling  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Sol. warm  $\text{H}_2\text{SO}_4 \rightarrow$  yellow col.; pptd. unchanged on dilution.

N-Acetyl: needles. M.p.  $125^\circ$ .

N-Benzoyl: needles. M.p.  $170^\circ$ .

N-1-Naphthoyl: needles. M.p.  $150^\circ$ .

N-2-Naphthoyl: needles. M.p.  $197-8^\circ$ .

N-p-Toluenesulphonyl: m.p.  $174^\circ$ .

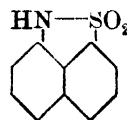
Rule, Brown, *J. Chem. Soc.*, 1934, 137.

Corbellini, Barbaro, *Giorn. chim. ind. applicata*, 1933, 15, 335.

I.G., D.R.P., 531,889, 511,212, (*Chem. Abstracts*, 1932, 26, 155; 1931, 25, 1266).

Pisovschi, *Bull. soc. chim.*, 1911, 9, 86.

### 1 : 8-Naphthasultam



$\text{C}_{10}\text{H}_7\text{O}_2\text{NS}$

MW, 205

Needles from  $\text{C}_6\text{H}_6$ . M.p.  $177-8^\circ$ . Sol.  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ ,  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ . Sols. fluor. green. Alc.  $\text{FeCl}_3 \rightarrow$  dark blue ppt.

N-Me:  $\text{C}_{11}\text{H}_9\text{O}_2\text{NS}$ . MW, 219. Yellowish needles from  $\text{MeOH}$ . M.p.  $125^\circ$ .

N-Et:  $\text{C}_{12}\text{H}_{11}\text{O}_2\text{NS}$ . MW, 233. M.p.  $85^\circ$ .

N-Phenyl: m.p.  $158^\circ$ .

N-p-Tolyl: m.p.  $152^\circ$ .

N-Acetyl: cryst. from  $\text{EtOH}$ . M.p.  $188^\circ$ . Sol.  $\text{CHCl}_3$ ,  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ .

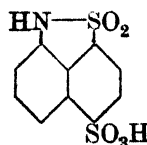
König, Wagner, *Ber.*, 1924, 57, 1056.

König, Köhler, *Ber.*, 1922, 55, 2146.

Dannert, *J. Am. Chem. Soc.*, 1907, 29, 1319.

Zincke, Jülicher, *Ann.*, 1916, 411, 202.

### 1 : 8-Naphthasultam-4-sulphonic Acid



$\text{C}_{10}\text{H}_7\text{O}_5\text{NS}_2$

MW, 285

Faintly pink needles. Very sol.  $\text{H}_2\text{O}$ .

*Mono-K salt*: prisms +  $1\frac{1}{2}\text{H}_2\text{O}$ . Decomp. at  $300^\circ$ . Sol. hot, spar. sol. cold  $\text{H}_2\text{O}$ .

*Di-K salt*: cryst. +  $1\text{H}_2\text{O}$ . Fluor. green. Stable at  $300^\circ$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH.

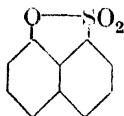
*Chloride*:  $\text{C}_{10}\text{H}_6\text{O}_4\text{NS}_2\text{Cl}$ . MW, 303.5. Leaflets. Decomp. above  $185^\circ$ . Sol.  $\text{C}_6\text{H}_6$ , toluene.

*Anilide*: colourless plates. Decomp. above  $230^\circ$ .

$\alpha$ -Naphthalide: leaflets. M.p.  $240^\circ$  decomp.

König, Keil, *Ber.*, 1922, 55, 2149.

**Naphthasultone** (1-Naphthol-8-sulphonolactone)



$\text{C}_{10}\text{H}_6\text{O}_3\text{S}$

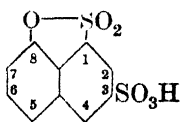
MW, 206

Stout glassy prisms from  $\text{C}_6\text{H}_6$ . M.p.  $156^\circ$ . Dist. undecomp. above  $360^\circ$ . Very sol.  $\text{CHCl}_3$ . Sol. hot  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH. Very spar. sol.  $\text{CS}_2$ . Insol.  $\text{H}_2\text{O}$ . Stable to cold alkalis.

Erdmann, *Ann.*, 1888, 247, 344.

Cumming, Muir, *Chem. Abstracts*, 1934, 28, 4410.

**Naphthasultone-3-sulphonic Acid** (1-Naphthol-8-sulphonolactone-3-sulphonic acid)



$\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2$

MW, 286

Long needles from  $\text{H}_2\text{O}$ . M.p.  $241^\circ$ .

*Na salt*: needles +  $3\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . Sol. 93 parts  $\text{H}_2\text{O}$  at  $15^\circ$ .

*Ba salt*: needles. Insol. cold  $\text{H}_2\text{O}$ .

Bernthsen, *Ber.*, 1889, 22, 3331.

B.D.C., E.P., 296,458 (derivs.).

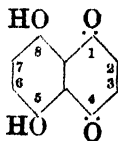
**Naphthasultone-4-sulphonic Acid** (1-Naphthol-8-sulphonolactone-4-sulphonic acid).

*Na salt*: leaflets or plates +  $3\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . More sol.  $\text{H}_2\text{O}$  than the 3-sulphonate (cf. above).

*Ba salt*: sol. cold  $\text{H}_2\text{O}$ .

Bernthsen, *Ber.*, 1890, 23, 3090.

**Naphthazarin** (5-8-Dihydroxy-1:4-naphthoquinone)



$\text{C}_{10}\text{H}_6\text{O}_4$

MW, 190

Reddish-brown needles with green reflex from EtOH. Sublimes in vac./2-10 mm. Decomp. on heating at ord. press. Mod. sol. AcOH. Spar. sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Sol. alkalis  $\rightarrow$  cornflower blue col. Gives coloured lakes with polyvalent metal hydroxides. Sol. conc.  $\text{H}_2\text{SO}_4$  with magenta col. Heat with NaOH.Aq.  $\rightarrow$  naphthapurpurin.

*Diacetate*: golden-yellow prisms from  $\text{CHCl}_3$ . M.p.  $192-3^\circ$ .

*Mono-phenylsemicarbazone*: m.p.  $218^\circ$  decomp.

*Di-phenylsemicarbazone*: m.p.  $285-7^\circ$  decomp. (darkens at  $280^\circ$ ).

*p-Bromophenylsemicarbazone*: decomp. at  $223^\circ$ .

*p-Nitrophenylsemicarbazone*: decomp. at  $234^\circ$ .

Ellis, Olpin, Kirk, U.S.P., 1,911,945, (*Chem. Abstracts*, 1933, 27, 3952).

Dreyfus, F.P., 667,917, (*Chem. Abstracts*, 1930, 24, 1393).

Zahn, Ochwat, *Ann.*, 1928, 462, 72.

Pfeiffer, *Ber.*, 1927, 60, 111.

Charrier, Tocco, *Gazz. chim. ital.*, 1923, 53, 431.

Wheeler, Edwards, *J. Am. Chem. Soc.*, 1916, 38, 387.

Friedländer, Silberstein, *Monatsh.*, 1902, 23, 518.

**o-Naphthazarin** (5:6-Dihydroxy-1:4-naphthoquinone).

Dark red needles with green metallic reflex from  $\text{H}_2\text{O}$ . M.p.  $201-2^\circ$ . Very sol.  $\text{Me}_2\text{CO}$ . Sol. EtOH. Spar. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol. ligroin. Sol. in NaOH.Aq.  $\rightarrow$  cornflower-blue  $\rightarrow$  green col. Sol. in conc.  $\text{H}_2\text{SO}_4$   $\rightarrow$  dull violet col. and gradually decomp.

*Mono-Py salt*: glittering green cryst. from Py.

Dimroth, Roos, *Ann.*, 1927, 456, 177, 186.

**Naphthazole.**

See  $\alpha$ -Naphthindole.

**Naphthenes.**

Cyclic (5- and 6-membered) saturated hydrocarbons of polymethylene type comprising about one-fourth of the total vol. of the world's crude petroleum oil. They include monocyclic hydrocarbons of from 4 up to 12 carbon atoms and polycyclic of 13 to 26 carbon atoms. They are comparatively stable, not reacting readily with conc.  $\text{H}_2\text{SO}_4$ , nitrating mixture, or halogens. Under the influence of heat or certain chemical reagents, or both combined, they may undergo according to circumstances such reactions as carbon-carbon scission, hydrogenation or dehydrogenation, polymerisation or depolymerisation, isomerisation. Their sulphonic acids are strongly capillary active. Among the simpler

naphthenes identified in crude oil are cyclobutane, cyclopentane, methylcyclopentane, cyclohexane, mono-, di- and tri-methylcyclohexanes, cycloheptane, cyclo-octane.

Sakhanow *et al.*, *Chem. Abstracts*, 1934, **28**, 295-9.

Dijk, *ibid.*, 886.

Naphthali, *Fettchem. Umschau*, 1933, **40**, 149, 176, 219 (Review).

Egloff, Bollmann, Levinson, *J. Phys. Chem.*, 1931, **35**, 3489 (Bibl., Review).

Petrov, *Chem. Abstracts*, 1930, **24**, 4415.

Komppa, *Ber.*, 1929, **62**, 1562.

### Naphthenic Acids.

Naphthenecarboxylic acids found in petroleum; one of the simplest has been shown to be 1-methylcyclopentane-carboxylic acid. Members of this series containing 9 and 10 carbon atoms in a monocyclic structure have been made artificially by the cracking of oleic acid in the presence of  $H_2O$  and alumina at  $380-90^\circ$ , or by air-oxidation of petroleum at  $90-100^\circ$ . Salts of naphthenic acids have wetting and emulsifying properties. They have also been used as disinfectants and in the resin and lacquer industries.

Malyatskii, Margolis, *Chem. Abstracts*, 1933, **27**, 4153.

Petrov, Ivanov, *J. Am. Chem. Soc.*, 1932, **54**, 239.

Braun *et al.*, *Ann.*, 1931, **490**, 100-79.

Carpenter, *Journal of the Institute of Petroleum Technology*, 1930, **16**, 284 (Review).

Komppa, *Ber.*, 1929, **62**, 1562.

Tiutiunnikov, *Chem. Abstracts*, 1927, **21**, 4055.

Augustin, *Seifensieder Zeitung*, 1927, **54**, 899.

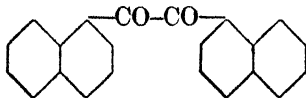
Zernik, *Chem. Zentr.*, 1925, **II**, 1403.

Tanaka, Nagai, *Chem. Abstracts*, 1926, **20**, 2744.

### Naphthidine.

See 4 : 4'-Diamino-1 : 1'-dinaphthyl.

$\alpha$ -Naphthil (1 : 1'-Dinaphthoyl)



$C_{22}H_{14}O_2$

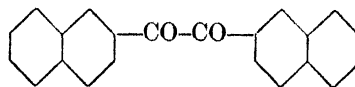
MW, 310

Yellow prisms from  $C_6H_6$ . M.p.  $189-90^\circ$ .

Quinoxaline deriv. : m.p.  $203-4^\circ$ . Conc.  $H_2SO_4$   $\rightarrow$  deep indigo-blue col.

Gomberg, Bachmann, *J. Am. Chem. Soc.*, 1930, **52**, 4972.

$\beta$ -Naphthil (2 : 2'-Dinaphthoyl)



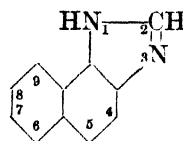
$C_{22}H_{14}O_2$

MW, 310

Cream-coloured needles from hot  $C_6H_6$ . M.p.  $157-8^\circ$ .

Gomberg, Bachmann, *J. Am. Chem. Soc.*, 1928, **50**, 2767.

$\alpha$  :  $\beta$ -Naphthiminazole



$C_{11}H_8N_2$

MW, 168

Leaflets from  $C_6H_6$ . M.p.  $178^\circ$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol.  $C_6H_6$ ,  $CHCl_3$ , ligroin.

1-N-Acetyl : needles from  $C_6H_6$ . M.p.  $153^\circ$ .

1-N-Benzoyl : needles from EtOH. M.p.  $120^\circ$ .

1-N-Me :  $C_{12}H_{10}N_2$ . MW, 182. Needles from Et<sub>2</sub>O or EtOH.Aq. M.p.  $88^\circ$ .

1-N-Et :  $C_{13}H_{12}N_2$ . MW, 196. Prisms from Et<sub>2</sub>O. M.p.  $129-30^\circ$ . Et<sub>2</sub>O sols. fluor. blue.

3-N-Et : yellow oil.  $HgCl_2$  double salt : m.p.  $182^\circ$ .

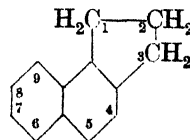
Fischer, Dietrich, Weiss, *J. prakt. Chem.*, 1920, **100**, 171.

Fischer, Reindl, Fezer, *Ber.*, 1901, **34**, 933.

### peri-Naphthiminazole.

See Perimidine.

$\alpha$ -Naphthindane (4 : 5-Benzhydrindene, 2 : 3-dihydronaphthindene)



$C_{13}H_{12}$

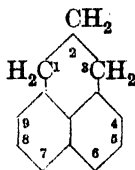
MW, 168

Colourless oil with faint aromatic odour. B.p.  $294-5^\circ/757$  mm.  $D_4^{20}$  1.066.  $n_D^{20}$  1.6290. Tends to resinify on exposure to light.  $Na_2Cr_2O_7 + AcOH \rightarrow \alpha$ -naphthindanone-3. Alk.  $K_3Fe(CN)_6 \rightarrow$  naphthalene-1 : 3-dicarboxylic acid.

Picrate : m.p.  $110^\circ$ .

Kruber, *Ber.*, 1932, **65**, 1383.

**peri-Naphthindane** (peri-Trimethylenenaphthalene)



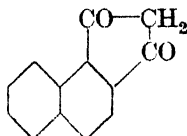
$C_{13}H_{12}$

MW, 168

Silky leaflets from dil. EtOH. M.p. 68–9°. Picrate : m.p. 134–5°.

Fleischer, Retze, *Ber.*, 1922, **55**, 3280.

$\alpha$  :  $\beta$ -Naphthindandione (4 : 5-Benzdiketohydrindene)



$C_{13}H_8O_2$

MW, 196

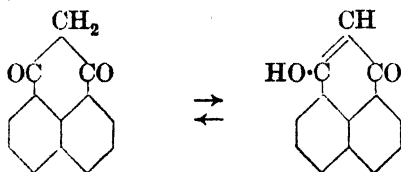
Yellow needles from EtOH. M.p. 180°. Sol. AcOH. Very spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Easily sol. alkalis with red col.

Benzylidene deriv. : m.p. 190°. Hyd. by hot alkalis into its components.

Di-phenylhydrazone : m.p. 220° decomp.

Noto, *Gazz. chim. ital.*, 1915, **45**, ii, 127, 427.

**peri-Naphthindandione** (1 : 8-Malonylnaphthalene)



$C_{13}H_8O_2$

MW, 196

Yellow or reddish-brown cryst. from AcOH. M.p. 265° decomp. (brown at 250°). Sol. EtOH, AcOH, weak bases, alkali carbonates. Very spar. sol. H<sub>2</sub>O, xylene, pet. ether.

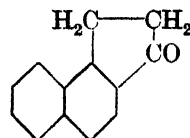
1-Me enol-ether :  $C_{14}H_{10}O_2$ . MW, 210. Yellowish plates. M.p. 144°.

1-Et enol-ether :  $C_{15}H_{12}O_2$ . MW, 224. Yellowish brown cryst. M.p. 148°.

Badische, D.R.P., 283,365, (*Chem. Zentr.*, 1915, I, 965).

Fleischer, Retze, *Ber.*, 1922, **55**, 3280.

$\alpha$ -Naphthindanone (4 : 5-Benzhydrindone)



$C_{13}H_{10}O$

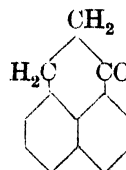
MW, 182

Colourless needles from MeOH or ligroin. M.p. 103°. Yellow sol. in conc. H<sub>2</sub>SO<sub>4</sub> with blue fluor.

I.G., D.R.P., 512,717, (*Chem. Abstracts*, 1931, **25**, 1260).

Mayer, Müller, *Ber.*, 1927, **60**, 2283.

**peri-Naphthindanone** (7 : 8-Dihydrophenalene-9)



$C_{13}H_{10}O$

MW, 182

Yellow leaflets. M.p. 85–6°. Unstable.

Oxime : needles from EtOH. M.p. 124–5°.

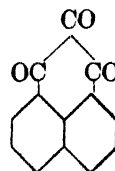
Benzylidene deriv. : yellow cryst. from MeOH. M.p. 163°.

Mayer, Sieglitz, *Ber.*, 1922, **55**, 1844.

Braun, Manz, Reinsch, *Ann.*, 1929, **468**, 301.

Cf. Cook, Hewitt, *J. Chem. Soc.*, 1934, 369, 373.

**peri-Naphthindantrione**



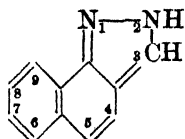
$C_{13}H_6O_3$

MW, 210

M.p. 273° decomp. Sol. xylene. Spar. sol. C<sub>6</sub>H<sub>6</sub>. In moist air forms hydrate C<sub>10</sub>H<sub>6</sub>(CO)<sub>2</sub>C(OH)<sub>2</sub>, golden-yellow prisms. Sol. 50 parts hot, 500 parts cold H<sub>2</sub>O. With o-phenylenediamine → a phenazine, yellow needles from C<sub>6</sub>H<sub>6</sub>, m.p. 255–6°.

Alcoholate : C<sub>10</sub>H<sub>6</sub>(CO)<sub>2</sub>C(OH)(OC<sub>2</sub>H<sub>5</sub>). MW, 256. Triclinic plates from EtOH. Decomp. at 140°.

Errera, *Gazz. chim. ital.*, 1913, **43**, i, 583.

$\alpha$ -Naphthindazole $C_{11}H_8N_2$ 

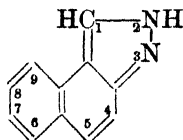
MW, 168

M.p. 158°.

2-N-Acetyl : m.p. 108-9°.

Picrate : m.p. 193°.

Vesely, Medvedeva, Müller, *J. Chem. Soc. Abstracts*, 1935, 991.

 $\beta$ -Naphthindazole $C_{11}H_8N_2$ 

MW, 168

M.p. 231°.

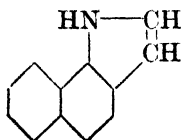
2-N-Acetyl : m.p. 116.5°.

Picrate : m.p. 217-18°.

See previous reference.

## Naphthindenone.

See Phenalone.

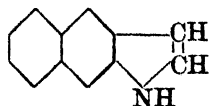
 $\alpha$ -Naphthindole (Naphthazole) $C_{12}H_9N$ 

MW, 167

Cryst. from MeOH. M.p. 173°.

Mayer, Oppenheimer, *Ber.*, 1918, 51, 1240.

## 2 : 3-Naphthindole

 $C_{12}H_9N$ 

MW, 167

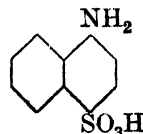
Cryst. M.p. 68-70°. Sol. ord. org. solvents.  
Pine-chip reaction  $\rightarrow$  violet col.

I.G., D.R.P., 516,675, (*Chem. Zentr.*, 1931, I, 1832).

## peri-Naphthindone.

See Phenalone.

## Naphthionic Acid (1-Naphthylamine-4-sulphonic acid)

 $C_{10}H_7O_3NS$ 

MW, 223

Needles +  $\frac{1}{2}H_2O$  from  $H_2O$ . Very spar. sol.  $H_2O$ . Sols. of the acid and its salts fluor. strongly blue.  $k = 2.1 \times 10^{-3}$  at 25°.  $NaHg \rightarrow$  1-naphthylamine +  $SO_2$ . The Na salt +  $SO_2$  in  $H_2O$  at 95°  $\rightarrow$  1-naphthol-4-sulphonic acid. Important intermediate for azo dyestuffs, e.g. Congo Red, Benzopurpurins, and Fast Reds.

N-Acetyl : needles. Sol.  $H_2O$ , EtOH. Forms series of arylamine salts of definite m.ps. *Et ester* : m.p. 148°. *Amide* : m.p. 241°. *Anilide* : m.p. 231°.

N-Me :  $C_{11}H_{11}O_3NS$ . MW, 237. Needles. Sol.  $H_2O$ . *Na salt* : needles. Very sol.  $H_2O$ .

N-Di-Me :  $C_{12}H_{13}O_3NS$ . MW, 251. Prisms +  $1H_2O$  from  $H_2O$ .

*Amide* :  $C_{10}H_{10}O_2N_2S$ . MW, 222. Needles from EtOH. M.p. 206°.

Langguth, *Chimie et Industrie*, 1930, 24, 31, (*Chem. Abstracts*, 1930, 24, 5035).

I.G., E.P., 326,022, (*Ibid.*, 4170).

Wahl, Vermeylen, *Bull. soc. chim.*, 1927, 41, 522; *Compt. rend.*, 1927, 184, 334.

Forster, Watson, *J. Soc. Chem. Ind.*, 1927, 46, 225t.

Ehrhardt, Hereward, E.P., 254,402, (*Chem. Abstracts*, 1927, 21, 2479).

Rupe, Metzger, *Helv. Chim. Acta*, 1925, 8, 842.

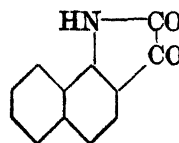
Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

Füssganger, *Ber.*, 1902, 35, 977.

Badische, D.R.P., 117,471, (*Chem. Zentr.*, 1901, I, 349).

Bretscheider, *J. prakt. Chem.*, 1897, 55, 299.

Paul, *Z. angew. Chem.*, 1896, 9, 685.

1 : 2-Naphthisatin ( $\alpha$ -Naphthisatin) $C_{12}H_7O_2N$ 

MW, 197



Red needles from EtOH. M.p. 255°. Spar. sol. EtOH. Very spar. sol. H<sub>2</sub>O. Green sol. in conc. H<sub>2</sub>SO<sub>4</sub>, on addn. of trace of thiophene → blue col.

*Phenylhydrazone* : m.p. 286° (262°).

I.G., E.P., 308,980, (*Chem. Zentr.*, 1930, I, 287).

I.G., E.P., 286,358, (*Chem. Abstracts*, 1929, 23, 156).

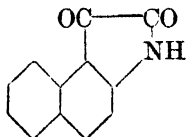
Stollé, *J. prakt. Chem.*, 1922, 105, 144.

Martinet, *Ann. chim.*, 1919, 11, 94, 118;

*Compt. rend.*, 1918, 166, 851; cf. *Chem. Zentr.*, 1919, III, 711.

Mayer, Oppenheimer, *Ber.*, 1918, 51, 1241, 1245.

### 2 : 1-Naphthisatin (*β*-Naphthisatin)



C<sub>12</sub>H<sub>7</sub>O<sub>2</sub>N

MW, 197

Red needles from EtOH. M.p. 253° (248°). Insol. H<sub>2</sub>O. Alc. KOH → dark violet col. Sol. in conc. H<sub>2</sub>SO<sub>4</sub> → intense reddish-brown col.

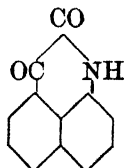
*Phenylhydrazone* : m.p. 225°.

N-Et : m.p. 173°. *Phenylhydrazone* : m.p. 180°.

Kränzlein, Wolfram, Hausdörfer, U.S.P., 1,792,170, (*Chem. Abstracts*, 1931, 25, 1845).

Wahl, Lobeck, *Ann. chim.*, 1929, 12, 156. See also second and third references above.

### 1 : 8-Naphthisatin (*peri*-Naphthisatin)



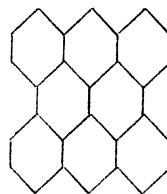
C<sub>12</sub>H<sub>7</sub>O<sub>2</sub>N

MW, 197

Cryst. from PhNO<sub>2</sub>. M.p. above 300°. Sol. in conc. H<sub>2</sub>SO<sub>4</sub> with reddish-yellow col. Sol. alkalis with intense red col.

I.G., Swiss P., 126,721, (*Chem. Zentr.*, 1929, II, 2104).

### meso-Naphthodianthrene



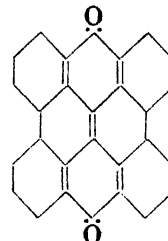
C<sub>28</sub>H<sub>14</sub>

MW, 350

Stout dark blue needles with violet cast from PhNO<sub>2</sub>. Sublimes. Very spar. sol. ord. org. solvents → blue sols. Conc. H<sub>2</sub>SO<sub>4</sub> → green-blue-violet col.

Scholl, Meyer, *Ber.*, 1934, 67, 1237.

### meso-Naphthodianthrene



C<sub>28</sub>H<sub>12</sub>O<sub>2</sub>

MW, 380

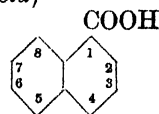
Glittering brown micro-leaflets from quinoline. Does not melt, but slowly decomp. at 550°.

Scholl, *Ber.*, 1919, 52, 1835.

### Naphthohydroquinone.

See 1 : 2- and 1 : 4-Dihydroxynaphthalenes.

**1-Naphthoic Acid** (*Naphthalene-1-carboxylic acid*, *α*-naphthoic acid)



C<sub>11</sub>H<sub>8</sub>O<sub>2</sub>

MW, 172

Needles from AcOH.Aq. M.p. 161°. Sol. EtOH. Spar. sol. H<sub>2</sub>O. Heat of comb. C<sub>p</sub> 1232.6 Cal., C<sub>v</sub> 1232.0 Cal. *k* = 2.04 × 10<sup>-4</sup> at 25°.

*Et ester* : C<sub>13</sub>H<sub>12</sub>O<sub>2</sub>. MW, 200. B.p. 309°, 220.5°/74 mm., 183.6°/20 mm. D<sub>15</sub><sup>20</sup> 1.1274.

*d-Amyl ester* : C<sub>16</sub>H<sub>18</sub>O<sub>2</sub>. MW, 242. B.p. 222°/25 mm. [α]<sub>D</sub><sup>20</sup> + 5.28°.

*Bornyl ester* : C<sub>21</sub>H<sub>24</sub>O<sub>2</sub>. MW, 308. M.p. 69-70°. [α]<sub>D</sub><sup>20</sup> - 33.5° in EtOH.

*l-Menthyl ester* : C<sub>21</sub>H<sub>26</sub>O<sub>2</sub>. MW, 310. B.p. 215°/20 mm. D<sub>15</sub><sup>20</sup> 1.0557. [α]<sub>D</sub><sup>20</sup> - 79.5° in EtOH.

*Chloride* : C<sub>11</sub>H<sub>7</sub>OCl. MW, 190.5. M.p. 20°. B.p. 297.5°, 172-3°/15 mm., 163°/10 mm.

*Amide* : C<sub>11</sub>H<sub>9</sub>ON. MW, 171. M.p. 202°.

*Anhydride*:  $C_{22}H_{14}O_3$ . MW, 326. M.p. 145°. *Nitrile*: see 1-Naphthonitrile. *Piperidide*: m.p. 85–7°.

Gilman, McCorkle, Calloway, *J. Am. Chem. Soc.*, 1934, **56**, 745.  
Salkind, *Ber.*, 1934, **67**, 1033.  
Gilman, St. John, Schulze, *Organic Syntheses*, 1931, **XI**, 80.  
McMaster, Langreck, *J. Am. Chem. Soc.*, 1917, **39**, 106.  
Whitmore, Fox, *J. Am. Chem. Soc.*, 1929, **51**, 3363.  
Loder, Whitmore, *J. Am. Chem. Soc.*, 1935, **57**, 2727.

**2-Naphthoic Acid** (*Naphthalene-2-carboxylic acid*,  $\beta$ -*naphthoic acid*).

Needles from ligroin, plates from  $Me_2CO$ . M.p. 184° (185.5°). Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol. ligroin, hot  $H_2O$ . Heat of comb.  $C_p$  1228.4 Cal.,  $C_v$  1227.8 Cal.  $k = 6.78 \times 10^{-5}$  ( $5.23 \times 10^{-5}$ ) at 25°.

*Me ester*:  $C_{12}H_{10}O_2$ . MW, 186. M.p. 77°. B.p. 290°.

*Et ester*: f.p. 32°. B.p. 308–9°, 224°/74 mm.  $D_4^{18}$  1.1212.

*d-Amyl ester*: b.p. 265°/about 100 mm.  $D_4^{20}$  1.0531.  $[\alpha]_D^{20} + 9.34^\circ$ .

*Bornyl ester*: m.p. 88–90°.  $[\alpha]_D^{20} - 34.5^\circ$  in EtOH.

*l-Menthyl ester*: m.p. 77–77.5°.  $[\alpha]_D^{20} - 98.5^\circ$  in  $C_6H_6$ .

*dl-Menthyl ester*: m.p. 70°.

*Neomenthyl ester*: m.p. 98°.

*Chloride*: m.p. 43°. B.p. 304–6°.

*Amide*: m.p. 192° (195°).

*Anhydride*: m.p. 133–4°.

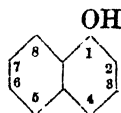
*Nitrile*: see 2-Naphthonitrile.

*Piperidide*: m.p. 88–90°.

Gilman, St. John, *Rec. trav. chim.*, 1929, **48**, 743.

Salkind, *Ber.*, 1934, **67**, 1033.

**1-Naphthol** (*1-Hydroxynaphthalene*,  $\alpha$ -*naphthol*)



$C_{10}H_8O$

MW, 144

F.p. 96.1° (94.2°). M.p. 94°. B.p. 278–80°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ .  $D_4^{20}$  1.09539.  $n_D^{20}$  1.62064. Sublimes. Heat of comb.  $C_p$  1188.5 Cal. Volatile in steam. Reduces Tollen's.  $FeCl_3 \rightarrow \alpha$ -dinaphthol.

Dict. of Org. Comp.—III.

$CrO_3$  in AcOH  $\rightarrow \alpha$ -naphthoquinone. Na + amyl alcohol  $\rightarrow ar$ -tetrahydro-1-naphthol.

*Chloroformyl*: b.p. 132°/5 mm.

*Acetyl*: m.p. 48–9°.

*Chloroacetyl*: m.p. 48°.

*Oxalyl*: m.p. 161–2°.

*Succinyl*: m.p. 155°.

*Benzoyl*: m.p. 56°.

3 : 5-Dinitrobenzoyl: m.p. 217.4°.

*Salicyloyl*: aliph. M.p. 83°.

*Sulphite*: m.p. 92–3°.

*Acid sulphate*: m.p. 182°.

*Di-acid phosphate*: m.p. 142°.

*Phosphate*: m.p. 145° (148–9°).

*Picrate*: m.p. 189°.

*Me ether*: see Methyl 1-naphthyl Ether.

*Et ether*: see Ethyl 1-naphthyl Ether.

*Propyl ether*:  $C_{13}H_{14}O$ . MW, 186. B.p. 298°/762 mm.  $D_4^{18}$  1.04471.  $n_D^{18}$  1.59277.

*Isoamyl ether*:  $C_{15}H_{18}O$ . MW, 214. B.p. 317–19°/741.9 mm.  $D_4^{18}$  1.00689.  $n_D^{18}$  1.57049.

$\alpha$ -*Glyceryl ether*:  $C_{13}H_{14}O_3$ . MW, 218. M.p. 95°.

$\alpha$  :  $\alpha$ -*Diglyceryl ether*:  $C_{23}H_{20}O_3$ . MW, 344. M.p. 116°.

*Phenyl ether*:  $C_{16}H_{12}O$ . MW, 220. M.p. 55–6°. B.p. 349.5°/753 mm.

*p-Nitrobenzyl ether*:  $C_{17}H_{13}O_3N$ . MW, 267. M.p. 140°.

1-*Naphthyl ether*: see 1 : 1'-Dinaphthyl Ether.

Cotton, U.S.P., 1,962,137, (*Chem. Abstracts*, 1934, **28**, 4748).

Vendelshtein, Shpinel, *Chem. Abstracts*, 1933, **27**, 5320.

Franzen, Kempf, *Ber.*, 1917, **50**, 103.

**2-Naphthol** (*2-Hydroxynaphthalene*,  $\beta$ -*naphthol*).

Plates. M.p. 123°. B.p. 285–6°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ , pet. ether. Sublimes. Volatile in steam. Heat of comb.  $C_p$  1190.3. Cal. Reduces Tollen's.  $FeCl_3 \rightarrow \beta$ -dinaphthol. Na + AcOH  $\rightarrow ac$ -tetrahydro-1-naphthol + *ar*-tetrahydro-2-naphthol.

*Formyl*: b.p. 117°/1.5 mm.  $D^{23}$  1.1554.  $n_D^{20}$  1.60932.

*Acetyl*: m.p. 70°.

*Propionyl*: m.p. 51°.

*Isobutyryl*: m.p. 43°.

*Isovaleryl*: b.p. 180–4°/20 mm.

*Oxalyl*: m.p. 191° (188–9°).

*Malonyl*: m.p. 146–7°.

*Succinyl*: m.p. 163°.

*Benzoyl*: m.p. 107°.

*o-Nitrobenzoyl*: m.p. 112°.

m-Nitrobenzoyl : m.p. 134°.

p-Nitrobenzoyl : m.p. 169°.

3 : 5-Dinitrobenzoyl : m.p. 210·2°.

Salicyloyl : betol. M.p. 95°.

Di-acid phosphate : m.p. 167°.

Acid phosphate : m.p. 147-8°.

Phosphate : m.p. 111°.

Picrate : m.p. 157°.

Me ether : see Methyl 2-naphthyl Ether.

Et ether : see Ethyl 2-naphthyl Ether.

Propyl ether :  $C_{13}H_{14}O$ . MW, 186. M.p. 39·5-40°. Picrate : m.p. 75°.

Isopropyl ether : m.p. 41°. Picrate : m.p. 92°.

Isobutyl ether :  $C_{14}H_{16}O$ . MW, 200. M.p. 33°. Picrate : m.p. 80-80·5°.

Isoamyl ether :  $C_{15}H_{18}O$ . MW, 214. M.p. 26°. B.p. 323-6°/759·3 mm. decomp.  $D_4^{20}$  1·01555.  $n_D^{20}$  1·57679. Picrate : m.p. 90·5-100°.

$\alpha$ -Glyceryl ether :  $C_{13}H_{14}O_5$ . MW, 218. M.p. 109-10°.

Phenyl ether :  $C_{16}H_{12}O$ . MW, 220. M.p. 46°. B.p. 335·5°/753 mm.

o-Nitrophenyl ether :  $C_{16}H_{11}O_3N$ . MW, 265. Prisms from MeOH. M.p. 58°.

2 : 4-Dinitrophenyl ether :  $C_{16}H_{10}O_5N_2$ . MW, 310. M.p. 95°.

p-Tolyl ether :  $C_{17}H_{14}O$ . MW, 234. M.p. 135°.

Benzyl ether :  $C_{17}H_{14}O$ . MW, 234. M.p. 99°.

p-Nitrobenzyl ether :  $C_{17}H_{13}O_3N$ . MW, 279. M.p. 106·5°.

1-Naphthyl ether : see 1 : 2'-Dinaphthyl Ether.

2-Naphthyl ether : see 2 : 2'-Dinaphthyl Ether.

Vorozhtov, Krasova, *Chem. Abstracts*, 1933, 27, 5321.

Franzen, Kempf, *Ber.*, 1917, 50, 104.

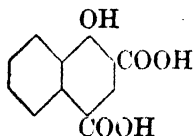
### Naphtholactone.

See under 8-Hydroxy-1-naphthoic Acid.

### Naphthol-carboxylic Acid.

See Hydroxynaphthoic Acid.

### 1-Naphthol-2 : 4-dicarboxylic Acid



$C_{12}H_8O_5$

MW, 232

Cryst. from EtOH. M.p. 304° decomp. Blue fluor. in EtOH.  $FeCl_3 \rightarrow$  green col.

Di-Me ester :  $C_{14}H_{12}O_5$ . MW, 260. M.p. 144°.

Di-Et ester :  $C_{16}H_{16}O_5$ . MW, 288. M.p. 98°.

Me ether :  $C_{13}H_{10}O_5$ . MW, 246. M.p. 252° decomp. Diamide :  $C_{13}H_{12}O_3N_2$ . MW, 244.

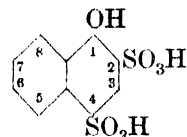
M.p. 198° decomp. Dianilide : m.p. 262° decomp.

Soc. anon. pour l'ind. chim. à Bâle, D.R.P., 373,736, (*Chem. Abstracts*, 1924, 18, 2174).

Montmollin, Spieler, U.S.P., 1,474,928, (*Chem. Abstracts*, 1924, 18, 693).

Menon, *J. Chem. Soc.*, 1935, 1061.

### 1-Naphthol-2 : 4-disulphonic Acid



$C_{10}H_8O_7S_2$

MW, 304

K salt sol. gives with  $FeCl_3$ , a blue col., with  $(CH_3COOK)$ , a red col. Heat with acids  $\rightarrow$  1-naphthol-2- and -4-sulphonic acids.

Dichloride :  $C_{10}H_6O_5S_2Cl_2$ . MW, 341. M.p. 149°.

Dianilide : m.p. 228°.

Bayer, D.R.P., 255,724. (*Chem. Zentr.*, 1913, I, 478).

Pollak, Gebauer-Fülneegg, Blumenstock-Halward, *Monatsh.*, 1928, 49, 193.

Conrad, Fischer, *Ann.*, 1893, 273, 105.

### 1-Naphthol-2 : 5-disulphonic Acid.

NaOH fusion  $\rightarrow$  1 : 5-dihydroxynaphthalene-2-sulphonic acid.

Bayer, D.R.P., 68,344.

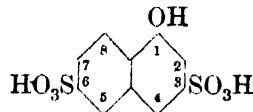
### 1-Naphthol-2 : 7-disulphonic Acid.

Zn salt sol.  $H_2O$ .  $HNO_3 \rightarrow$  2 : 4-dinitro-1-naphthol-7-sulphonic acid.

Vignon, D.R.P., 32,291.

Friedländer, Taussig, *Ber.*, 1897, 30, 1463.

### 1-Naphthol-3 : 6-disulphonic Acid



$C_{10}H_8O_7S_2$

MW, 304

Sol.  $H_2O$ .  $FeCl_3 \rightarrow$  blue col. Alkali salt sols. have green fluor.

Dichloride : carboxyl deriv., m.p. 95°.

Gebauer-Fuelneegg, Haemmerle, *J. Am. Chem. Soc.*, 1931, 53, 2653.

Gürke, Rudolph, D.R.P., 38,281.

### 1-Naphthol-3 : 7-disulphonic Acid.

Heat with  $NH_3 + NH_4Cl \rightarrow$  3-amino-1-

naphthol-7-sulphonic acid + 1 : 3-naphthalenediamine-7-sulphonic acid.

Freund, D.R.P., 27,346.

Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

**1-Naphthol-3 : 8-disulphonic Acid** ( $\epsilon$ -Acid, *epsilon acid*).

K salt sol.  $\longrightarrow$  blue col. with  $\text{FeCl}_3$ .

Na salt : prisms +  $6\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ .

Dichloride : *carbethoxyl deriv.*, m.p.  $180-1^\circ$ .

8-Amide :  $\text{C}_{10}\text{H}_9\text{O}_6\text{S}_2\text{N}$ . MW, 303. Cryst. Sol.  $\text{H}_2\text{O}$ .

B.D.C., F.P., 653,595, (*Chem. Abstracts*, 1929, 23, 3816); E.P., 296, 458.

Gebauer-Fuelnegg, Haemmerle, *J. Am. Chem. Soc.*, 1931, 53, 2653.

Kalle, D.R.P., 64,979.

**1-Naphthol-4 : 7-disulphonic Acid.**

Dichloride : *carbethoxyl deriv.*, m.p.  $120^\circ$ .

Friedländer, Taussig, *Ber.*, 1897, 30, 1460.

Gebauer-Fuelnegg, Haemmerle, *J. Am. Chem. Soc.*, 1931, 53, 2652.

Oehler, D.R.P., 74,744.

**1-Naphthol-4 : 8-disulphonic Acid** ( $\alpha$  Naphthol-disulphonic acid-S, *Schöllkopf's Acid*).

$\text{HNO}_3 \longrightarrow$  2 : 4-dinitro-1-naphthol-8-sulphonic acid. NaOH fusion  $\longrightarrow$  1 : 8-dihydroxynaphthalene-4-sulphonic acid.

Dichloride : *carbethoxyl deriv.*, m.p.  $177-9^\circ$ .

Gebauer-Fuelnegg, Haemmerle, *J. Am. Chem. Soc.*, 1931, 53, 2653.

Bernthsen, *Ber.*, 1890, 23, 3092.

Bucherer, *J. prakt. Chem.*, 1904, 69, 80; 1904, 70, 347.

**1-Naphthol-5 : 8-disulphonic Acid.**

Alk. fusion  $\longrightarrow$  1 : 8-dihydroxynaphthalene-4-sulphonic acid.

Bayer, D.R.P., 70,857.

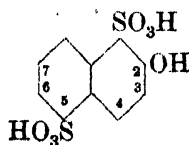
**1-Naphthol-6 : 8-disulphonic Acid.**

$\text{FeCl}_3 \longrightarrow$  green col. Alk. fusion  $\longrightarrow$  1 : 8-dihydroxynaphthalene-3-sulphonic acid.

Kalle, D.R.P., 82,563.

Bucherer, *J. prakt. Chem.*, 1904, 70, 347.

**2-Naphthol-1 : 5-disulphonic Acid**



$\text{C}_{10}\text{H}_8\text{O}_7\text{S}_2$

MW, 304

Dichloride :  $\text{C}_{10}\text{H}_6\text{O}_5\text{S}_2\text{Cl}_2$ . MW, 341. M.p.  $177^\circ$ .

Dianilide : m.p.  $231^\circ$ .

Pollak, Gebauer-Fuelnegg, Blumenstock-Halward, *Monatsh.*, 1929, 53 & 54, 83.

**2-Naphthol-1 : 6-disulphonic Acid.**

Cryst. +  $1\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .  $\text{FeCl}_3 \longrightarrow$  red col. Acids at  $90^\circ \longrightarrow$  2-naphthol-6-sulphonic acid.

Dichloride : m.p.  $111^\circ$ .

Dianilide : m.p.  $191^\circ$ .

Et ether : diamide,  $\text{C}_{12}\text{H}_{14}\text{O}_5\text{N}_2\text{S}_2$ . MW, 330. M.p.  $253-4^\circ$ .

Engel, *J. Am. Chem. Soc.*, 1930, 52, 2842.

Lapworth, *Chem. News*, 1895, 71, 206.

**2-Naphthol-1 : 7-disulphonic Acid.**

Salt sols. show blue fluor. Ba salt sol.  $\text{H}_2\text{O}$ . Heat with dil. HCl  $\longrightarrow$  2-naphthol-7-sulphonic acid.

p-Toluidine salt : m.p.  $219^\circ$ .

Dichloride : m.p.  $169^\circ$ .

Dianilide : m.p.  $233^\circ$ .

Bayer, D.R.P., 77,596.

Pollak, Gebauer-Fuelnegg, Blumenstock-Halward, *Monatsh.*, 1929, 53 & 54, 83.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, 46, 28r.

**2-Naphthol-3 : 6-disulphonic Acid** (R-Acid).

Needles. Sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ . Salt sols. show bluish-green fluor. Alk. fusion  $\longrightarrow$  2 : 3-dihydroxynaphthalene-6-sulphonic acid. The disodium salt (R Salt) is used extensively in the manufacture of azo dyestuffs.

Ba salt : spar. sol.  $\text{H}_2\text{O}$ . Insol. EtOH.

Aniline salt : m.p.  $254^\circ$ .

p-Chloroaniline salt : m.p.  $254^\circ$ .

m-Nitroaniline salt : m.p.  $299^\circ$ .

o-Toluidine salt : m.p.  $257^\circ$ .

m-Toluidine salt : m.p.  $242^\circ$ .

p-Toluidine salt : m.p.  $250^\circ$ .

m-Xylidine salt : m.p.  $196^\circ$ .

p-Cumidine salt : m.p.  $301^\circ$ .

1-Naphthylamine salt : m.p.  $292^\circ$ .

2-Naphthylamine salt : m.p.  $304^\circ$ .

p-Anisidine salt : m.p.  $267^\circ$ .

Dianisidine salt : m.p.  $317-18^\circ$ .

p-Phenetidine salt : m.p.  $244^\circ$ .

Dichloride :  $\text{C}_{10}\text{H}_6\text{O}_5\text{S}_2\text{Cl}_2$ . MW, 341. Carbethoxyl deriv., m.p.  $125^\circ$ .

Anilide : m.p.  $202^\circ$ .

*Et ether* : dichloride,  $C_{12}H_{10}O_5Cl_2S_2$ . MW, 369. M.p. 121°.

Pollak, Blumenstock-Halward, *Monatsh.*, 1928, **49**, 209.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, **46**, 277.

Kawaguchi, *Chem. Abstracts*, 1924, **18**, 2891.

Vorontzov, Sokolova, *Chem. Abstracts*, 1934, **28**, 3730.

Shcherbachev, Bashkistrova, *ibid.*, 5370.

Masters, E.P., 210,120, (*Chem. Abstracts*, 1924, **18**, 1673).

### 2-Naphthol-3 : 7-disulphonic Acid.

Salt sols. show green fluor. Alk. fusion  $\rightarrow$  2 : 7-dihydroxynaphthalene-3-sulphonic acid.

*Ba salt* : spar. sol.  $H_2O$ .

Bayer, D.R.P., 78,569.

Shcherbachev, Bashkistrova, *Chem. Abstracts*, 1934, **28**, 5370.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, **46**, 287.

### 2-Naphthol-4 : 7-disulphonic Acid.

Heated with aniline and aniline hydrochloride  $\rightarrow$  N:N'-diphenyl-1 : 3-naphthylenediamine-6-sulphonic acid.

Bayer, D.R.P., 77,860.

### 2-Naphthol-4 : 8-disulphonic Acid ( $\beta$ -Naphtholdisulphonic acid-C).

Sol.  $H_2O$ . Salt sols. show blue fluor.

Cassella, D.R.P., 65,997.

### 2-Naphthol-6 : 8-disulphonic Acid (G-Acid, $\beta$ -naphthol- $\gamma$ -disulphonic acid).

The disodium salt (G Salt) is used as intermediate in the manufacture of azo dyestuffs.

*o*-Chloroaniline salt : m.p. 255°.

*p*-Chloroaniline salt : m.p. 242°.

*o*-Toluidine salt : m.p. 270-1°.

*p*-Toluidine salt : m.p. 294°.

$\phi$ -Cumidine salt : m.p. 300-3°.

*p*-Phenetidine salt : m.p. 214-15°.

Dianisidine salt : m.p. 317°.

1-Naphthylamine salt : m.p. 302°.

2-Naphthylamine salt : m.p. 254°.

Dichloride : m.p. 161-2°. Carbethoxyl deriv., m.p. 131°.

Dianilide : m.p. 195°. Carbethoxyl deriv., m.p. 178°.

*Et ether* : dichloride,  $C_{12}H_{10}O_5Cl_2S_2$ . MW, 369. M.p. 158°.

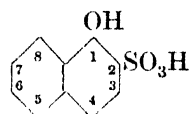
Masters, E.P., 210,120, (*Chem. Abstracts*, 1924, **18**, 1673).

Pollak, Blumenstock-Halward, *Monatsh.*, 1928, **49**, 208.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, **46**, 277.

Kawaguchi, *Chem. Abstracts*, 1924, **18**, 2891.

### 1-Naphthol-2-sulphonic Acid



$C_{10}H_8O_4S$

MW, 224

Plates from  $H_2O$ . Does not melt below 250°. Sol. hot  $H_2O$ . Insol.  $Et_2O$ . K salt  $\rightarrow$  blue col. with  $FeCl_3$ .

Chloride : acetyl deriv., m.p. 87.5°. Carboethoxyl deriv., m.p. 130°.

Anilide : acetyl deriv., m.p. 157.5°.

*p*-Toluidide : acetyl deriv., m.p. 135.5°.

Anschutz, *Ann.*, 1918, **415**, 64.

Bayer, F.P., 429,999, (*Chem. Abstracts*, 1912, **6**, 2536).

Gebauer-Fülneegg, Gluckmann, *Monatsh.*, 1929, **53** & **54**, 104.

### 1-Naphthol-3-sulphonic Acid.

Chloride : carboethoxyl deriv., m.p. 140°.

Anilide : m.p. 236°. Carboethoxyl deriv., m.p. 153°.

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, **I**, 478).

Gebauer-Fülneegg, Gluckmann, *Monatsh.*, 1929, **53** & **54**, 105.

### 1-Naphthol-4-sulphonic Acid (Nevile and Winthers' acid, NW-Acid).

Plates from  $H_2O$ . M.p. 170° decomp. (rapid heat). Sol.  $H_2O$ . Salt sols.  $\rightarrow$  blue col. with  $FeCl_3$ .  $K_2Cr_2O_7 \rightarrow \alpha$ -naphthoquinone. Intermediate for azo dyestuffs.

Aniline salt : m.p. 186-7°.

*p*-Chloroaniline salt : m.p. 184-5°.

*o*-Toluidine salt : m.p. 203-4°.

*p*-Toluidine salt : m.p. 196°.

*p*-Nitro-*o*-toluidine salt : m.p. 249°.

*m*-Xylidine salt : m.p. 228-9°.

$\phi$ -Cumidine salt : m.p. 227-8°.

Tolidine salt : m.p. 214-15°.

*o*-Anisidine salt : m.p. 202-3°.

*p*-Anisidine salt : m.p. 224°.

Dianisidine salt : m.p. 207-9°.

1-Naphthylamine salt : m.p. 216-17°.

Chloride : carboethoxyl deriv., m.p. 84°.

Anilide : m.p. 199-200°. Carboethoxyl deriv., m.p. 149°.

*Diphenylamide*: m.p. 176°.

*2-Naphthalide*: m.p. 204°.

*Et ether*: *Me ester*:  $C_{13}H_{14}O_4S$ . MW, 266. M.p. 105–6°. *Et ester*:  $C_{14}H_{16}O_4S$ . MW, 280. M.p. 102–3°. *Chloride*:  $C_{12}H_{11}O_3ClS$ . MW, 270.5. M.p. 101°. *Amide*:  $C_{12}H_{13}O_3NS$ . MW, 251. M.p. 167°.

Major, E.P., 328,220, (*Chem. Abstracts*, 1930, **24**, 5509).

Binns, Lurie, U.S.P., 1,880,701, (*Chem. Abstracts*, 1933, **27**, 515).

Gebauer-Fülneegg, Schlesinger, *Ber.*, 1928, **61**, 781.

Baddeley, Payman, Bainbridge, U.S.P., 1,452,481, (*Chem. Abstracts*, 1923, **17**, 1969); E.P. 186,515.

Rowe, J. *Soc. Dyers Colourists*, 1919, **35**, 128.

Zincke, Ruppertsberg, *Ber.*, 1915, **48**, 122.

Vorobjoff, Karlaseh, *Revue des matières colorantes, teinture, impression, blanchiment, apprêts*, 1935, **39**, 373.

### 1-Naphthol-5-sulphonic Acid.

Cryst. M.p. 110–12°.

*Chloride*: *acetyl deriv.*, m.p. 129°. *Carbethoxyl deriv.*, m.p. 174°.

*Anilide*: m.p. 201°. *Carbethoxyl deriv.*, m.p. 127°.

Gebauer-Fülneegg, Gluckmann, *Monatsh.*, 1929, **53** & **54**, 105.

Heller, *J. prakt. Chem.*, 1929, **121**, 196.

### 1-Naphthol-6-sulphonic Acid.

*Chloride*: *carbethoxyl deriv.*, m.p. 112°.

*Anilide*: m.p. 181°. *Carbethoxyl deriv.*, m.p. 140°.

Bayer, D.R.P., 109,122, (*Chem. Zentr.*, 1900, II, 359).

Gebauer-Fülneegg, Gluckmann, *Monatsh.*, 1929, **53** & **54**, 105.

### 1-Naphthol-7-sulphonic Acid.

Hygroscopic cryst. Sol.  $H_2O$ , EtOH.  $FeCl_3$  → brownish-violet col.

*Chloride*: *carbethoxyl deriv.*, m.p. 105°.

*Anilide*: m.p. 155°.

Gebauer-Fülneegg, Gluckmann, *Monatsh.*, 1929, **53** & **54**, 105.

See also first reference above.

### 1-Naphthol-8-sulphonic Acid ( $\alpha$ -Naphthol-sulphonic acid-S).

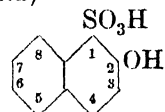
Cryst. +  $1H_2O$ . M.p. 106–7°. Mod. sol.  $H_2O$ .  $FeCl_3$  → green col.

*Amide*: m.p. 222° decomp.

Straub, Schneider, U.S.P., 1,503,172, (*Chem. Abstracts*, 1924, **18**, 2967).

Soc. anon. pour l'ind. chim. à Bâle, E.P., 207,162, (*Chem. Abstracts*, 1924, **18**, 1206).

### 2-Naphthol-1-sulphonic Acid (*Oxy-Tobias acid*, *Stebbin's acid*)



$C_{10}H_8O_4S$  MW, 224

Sol.  $H_2O$ .  $FeCl_3$  → red col. (blue with salt sols.).

*Aniline salt*: m.p. 182°.

*o-Toluidine salt*: m.p. 178–9°.

*p-Toluidine salt*: m.p. 162°.

*Chloride*:  $C_{10}H_7O_3ClS$ . MW, 242.5. M.p. 124°. *Acetyl deriv.*: m.p. 115.5°.

*Et ether*: *chloride*,  $C_{12}H_{11}O_3ClS$ . MW, 270.5. M.p. 115–16°. *Amide*:  $C_{12}H_{13}O_3NS$ . MW, 251. M.p. 158°.

Engel, *J. Am. Chem. Soc.*, 1930, **52**, 2841.

Tinker, Hanson, U.S.P., 1,934,216, (*Chem. Abstracts*, 1934, **28**, 495).

Anschutz, *Ann.*, 1918, **415**, 89.

Cotton, U.S.P., 1,913,748, (*Chem. Abstracts*, 1933, **27**, 4248).

Parmelee, U.S.P., 1,716,082, (*Chem. Abstracts*, 1929, **23**, 3716).

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, **46**, 29r.

### 2-Naphthol-3-sulphonic Acid.

Cryst. +  $1H_2O$ . Salt sols. show blue fluor.  $FeCl_3$  → deep blue col.

*Na salt*: cryst. +  $1H_2O$  from EtOH.

*Aniline salt*: m.p. 241–2°.

*1-Naphthylamine salt*: m.p. 247–8°.

*Amide*:  $C_{10}H_9O_3NS$ . MW, 223. M.p. 110°.

*Anilide*: m.p. 112°.

*Me ether*: *chloride*,  $C_{11}H_9O_3ClS$ . MW, 256.5. M.p. 137–8°. *Amide*:  $C_{11}H_{11}O_3NS$ . MW, 237. M.p. 113°. *Anilide*: m.p. 173–4°.

Holt, Mason, *J. Soc. Dyers Colourists*, 1930, **46**, 270.

### 2-Naphthol-4-sulphonic Acid.

Sol.  $H_2O$  with bluish-violet fluor.  $PCl_5$  → 1:3-dichloronaphthalene.

*Ba salt*: sol.  $H_2O$ .

Morgan, Jones, *J. Soc. Chem. Ind.*, 1923, **42**, 97r.

Bogdanov, Levkoev, Durmashkina, *Chem. Abstracts*, 1934, **28**, 4728.

## 2-Naphthol-5-sulphonic Acid

**2-Naphthol-5-sulphonic Acid** ( $\beta$ -Naphthol- $\gamma$ -sulphonic acid).

Na salt sol. shows greenish-blue fluor.  $\rightarrow$  violet-red col. with  $\text{FeCl}_3$ . KOH fusion  $\rightarrow$  1:6-dihydroxynaphthalene.

Armstrong, Wynne, *Chem. News*, 1889, **59**, 141.

Claus, *J. prakt. Chem.*, 1889, **39**, 315.

Dahl, D.R.P., 29,084.

**2-Naphthol-6-sulphonic Acid** (Schäffer Acid,  $\beta$ -naphtholsulphonic acid-S).

Cryst. +  $1\text{H}_2\text{O}$ . from  $\text{H}_2\text{O}$ , m.p.  $129^\circ$ ; +  $2\text{H}_2\text{O}$ , m.p.  $118^\circ$ . M.p.  $167^\circ$  anhyd. Sol.  $\text{H}_2\text{O}$ , EtOH. Na salt sol.  $\rightarrow$  green col. with  $\text{FeCl}_3$ . Intermediate for azo dyestuffs.

Aniline salt: m.p.  $264^\circ$ .

o-Chloroaniline salt: m.p.  $225^\circ$ .

p-Chloroaniline salt: m.p.  $234^\circ$ .

o-Toluidine salt: m.p.  $208^\circ$ .

p-Toluidine salt: m.p.  $248^\circ$ .

m-Xylidine salt: m.p.  $220^\circ$ .

$\psi$ -Cumidine salt: m.p.  $226-7^\circ$ .

p-Anisidine salt: m.p.  $230-1^\circ$ .

p-Phenetidine salt: m.p.  $255^\circ$ .

Dianisidine salt: m.p.  $282^\circ$ .

1-Naphthylamine salt: m.p.  $254^\circ$ .

2-Naphthylamine salt: m.p.  $268^\circ$ .

Phenyl ester:  $\text{C}_{16}\text{H}_{12}\text{O}_4\text{S}$ . MW, 300. M.p.  $131^\circ$ .

Chloride: acetyl deriv., m.p.  $107^\circ$  ( $95^\circ$ ). Carbethoxyl deriv., m.p.  $118^\circ$ .

Amide:  $\text{C}_{10}\text{H}_9\text{O}_3\text{NS}$ . MW, 223. M.p.  $237-9^\circ$ .

Di-Me amide:  $\text{C}_{12}\text{H}_{13}\text{O}_3\text{NS}$ . MW, 251. M.p.  $125^\circ$ .

Anilide: m.p.  $161^\circ$ . Carbethoxyl deriv., m.p.  $130^\circ$ .

Me ether: chloride,  $\text{C}_{11}\text{H}_9\text{O}_3\text{ClS}$ . MW, 256.5. M.p.  $93^\circ$ . Amide:  $\text{C}_{11}\text{H}_{11}\text{O}_3\text{NS}$ . MW, 237. M.p.  $199^\circ$ .

Et ether: chloride,  $\text{C}_{12}\text{H}_{11}\text{O}_3\text{ClS}$ . MW, 270.5. M.p.  $107.5^\circ$ . Amide:  $\text{C}_{12}\text{H}_{13}\text{O}_3\text{NS}$ . MW, 251. M.p.  $185^\circ$ .

Heller, *J. prakt. Chem.*, 1929, **121**, 196.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, **46**, 29r.

Wend, *Zeitschrift für Farbenindustrie*, 1929, **20**, 272.

Engel, *J. Am. Chem. Soc.*, 1930, **52**, 211, 2841.

Vorontzov, Sokolova, *Chem. Abstracts*, 1934, **28**, 3730.

Vorontzov, *Chem. Abstracts*, 1931, **25**, 5515.

**2-Naphthol-7-sulphonic Acid** (F Acid,  $\beta$ -naphthol- $\delta$ -sulphonic acid).

## 22 2-Naphthol-1:3:6:7-tetrasulphonic Acid

Cryst. +  $1\text{H}_2\text{O}$ , m.p.  $108-9^\circ$ ; +  $2\text{H}_2\text{O}$ , m.p.  $95^\circ$ ; +  $4\text{H}_2\text{O}$ , m.p.  $67^\circ$ ; anhyd. m.p.  $115-16^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Na salt sol. shows blue fluor.  $\rightarrow$  blue col. with  $\text{FeCl}_3$ .

Aniline salt: m.p.  $249^\circ$ .

p-Toluidine salt: m.p.  $237^\circ$ .

Et ether: chloride, m.p.  $103^\circ$ . Amide: m.p.  $172^\circ$ .

Harland, Forrester, Pain, *J. Soc. Chem. Ind.*, 1931, **50**, 100r.

Kogan, *Chem. Abstracts*, 1933, **27**, 978.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, **46**, 29r.

**2-Naphthol-8-sulphonic Acid** (Crocein acid, Bayer acid,  $\beta$ -naphthol- $\alpha$ -sulphonic acid).

Intermediate for azo dyestuffs.

Aniline salt: m.p.  $240^\circ$ .

o-Toluidine salt: m.p.  $242^\circ$ .

p-Toluidine salt: m.p.  $232^\circ$ .

1-Naphthylamine salt: m.p.  $241^\circ$ .

2-Naphthylamine salt: m.p.  $247-8^\circ$ .

Benzidine salt: m.p.  $293-4^\circ$  decomp.

Chloride: acetyl deriv., m.p.  $129^\circ$ . Carbethoxyl deriv., m.p.  $118^\circ$ .

Anilide: m.p.  $195^\circ$ .

Me ether: chloride,  $\text{C}_{11}\text{H}_9\text{O}_3\text{ClS}$ . MW, 256.5. M.p.  $137^\circ$ . Amide:  $\text{C}_{11}\text{H}_{11}\text{O}_3\text{NS}$ . MW, 237. M.p.  $153^\circ$ .

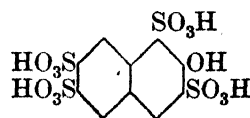
Et ether: chloride,  $\text{C}_{12}\text{H}_{11}\text{O}_3\text{ClS}$ . MW, 270.5. M.p.  $93^\circ$ . Amide:  $\text{C}_{12}\text{H}_{13}\text{O}_3\text{NS}$ . MW, 251. M.p.  $165^\circ$ .

Engel, *J. Am. Chem. Soc.*, 1930, **52**, 2841.

Gebauer-Fülneegg, Schlesinger, *Ber.*, 1928, **61**, 784.

Forster, Keyworth, *J. Soc. Chem. Ind.*, 1927, **46**, 29r.

**2-Naphthol-1:3:6:7-tetrasulphonic Acid**



$\text{C}_{10}\text{H}_8\text{O}_{13}\text{S}_4$

MW, 464

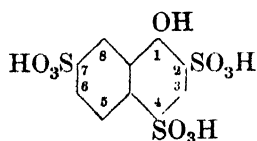
Alk. salt sols. show bluish-green fluor. Heat with  $\text{NH}_3 \rightarrow$  2-naphthylamine-1:3:6:7-tetrasulphonic acid. Hot dil. HCl  $\rightarrow$  2-naphthol-3:6:7-trisulphonic acid.

Na salt: sol.  $\text{H}_2\text{O}$ .

Ba salt: spar. sol.  $\text{H}_2\text{O}$ .

Anschutz, *Ann.*, 1918, **415**, 95.

## 1-Naphthol-2 : 4 : 7-trisulphonic Acid

 $C_{10}H_6O_{10}S_3$ 

MW, 384

Needles. Na salt sol.  $\rightarrow$  blue col. with  $FeCl_3$ . Ox.  $\rightarrow$  phthalic acid. Red. with sodium amalgam + acid  $\rightarrow$  1-naphthol-2 : 7-disulphonic acid.

Chloride :  $C_{10}H_5O_7Cl_3S_3$ . MW, 439.5. M.p.  $174^\circ$ .

Anilide : m.p.  $227^\circ$  ( $240^\circ$  decomp.).

Friedländer, Taussig, *Ber.*, 1897, **30**, 1463.

Badische, D.R.P., 10,785.

Gebauer-Fülneegg, Gluckmann, *Monatsh.*, 1929, **53** & **54**, 108.

Bender, *Ber.*, 1889, **22**, 993.

Gebauer-Fuelneegg, Haemmerle, *J. Am. Chem. Soc.*, 1931, **53**, 2651.

## 1-Naphthol-2 : 4 : 8-trisulphonic Acid.

Na salt sol.  $\rightarrow$  deep blue col. with  $FeCl_3$ . NaOH fusion  $\rightarrow$  1 : 8-dihydroxynaphthalene-2 : 4-disulphonic acid.

Dressel, Kothe, *Ber.*, 1894, **27**, 2144.

## 1-Naphthol-3 : 6 : 8-trisulphonic Acid (Oxy-Koch Acid).

KOH fusion at  $200^\circ \rightarrow$  1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid.

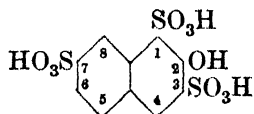
M.L.B., D.R.P., 71,495.

## 1-Naphthol-4 : 6 : 8-trisulphonic Acid.

Trichloride :  $C_{10}H_5O_7Cl_3S_3$ . MW, 439.5. M.p.  $217^\circ$ .

Gebauer-Fuelneegg, Haemmerle, *J. Am. Chem. Soc.*, 1931, **53**, 2651.

## 2-Naphthol-1 : 3 : 7-trisulphonic Acid

 $C_{10}H_6O_{10}S_3$ 

MW, 384

Alk. salt sols. show bluish-green fluor., and give violet col. with  $FeCl_3$ .  $NH_3 + NH_4Cl$  on heating  $\rightarrow$  2-naphthylamine-1 : 3 : 7-trisulphonic acid.

Na salt : sol.  $H_2O$ . Insol. EtOH.

Ba salt : sol.  $H_2O$ .

Dressel, Kothe, *Ber.*, 1894, **27**, 1207.

## 2-Naphthol-3 : 6 : 7-trisulphonic Acid.

Alk. salt sols. show bluish-green fluor., and with  $FeCl_3 \rightarrow$  violet col. Alk. fusion  $\rightarrow$  2 : 7-dihydroxynaphthalene-3 : 6-disulphonic acid. Heat with  $NH_3 \rightarrow$  2-naphthylamine-3 : 6 : 7-trisulphonic acid.

Na salt : spar. sol.  $H_2O$ .

Ba salt : spar. sol.  $H_2O$ .

Dressel, Kothe, *Ber.*, 1894, **27**, 1209.

Bayer, D.R.P., 78,569.

## 2-Naphthol-3 : 6 : 8-trisulphonic Acid.

Na salt sol. shows green fluor.  $\rightarrow$  deep violet col. with  $FeCl_3$ . Heat with  $NH_3 \rightarrow$  2-naphthylamine-3 : 6 : 8-trisulphonic acid.

Trifluoride :  $C_{10}H_5O_7F_3S_3$ . MW, 390. M.p.  $153-9^\circ$ .

Trichloride :  $C_{10}H_5O_7Cl_3S_3$ . MW, 439.5. M.p.  $196^\circ$ .

Trianilide : m.p.  $152-5^\circ$ .

Levinstein, *Ber.*, 1883, **16**, 462.

M.L.B., D.R.P., 22,038.

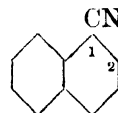
Blumenstock-Halward, Jusa, *Monatsh.*, 1928, **50**, 128.

Pollak, Gebauer-Fülneegg, Blumenstock-Halward, *Monatsh.*, 1928, **49**, 200.

## Naphthol Yellow S.

See Flavianic Acid.

## 1-Naphthonitrile (1-Cyanonaphthalene)

 $C_{11}H_7N$ 

MW, 153

Needles from ligroin. M.p.  $37.5^\circ$  ( $35-6^\circ$ ,  $33.5^\circ$ ). B.p.  $299^\circ$  ( $297.8^\circ$ ,  $296.5^\circ$ ). Sol. EtOH.  $D_{15}^{25}$  1.1167.  $n_D^{17.5}$  1.6298. Heat of comb.  $C_p$  1333.2 Cal.

Maihle, *Bull. soc. chim.*, 1918, **23**, 237.

Merck, D.R.P., 168,728, (*Chem. Zentr.*, 1906, I, 1469).

McRae, *J. Am. Chem. Soc.*, 1930, **52**, 4550.

Rule, Barnett, *J. Chem. Soc.*, 1932, 177.

## 2-Naphthonitrile (2-Cyanonaphthalene).

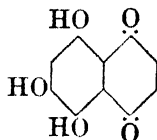
Leaflets from ligroin. M.p.  $66^\circ$  ( $63^\circ$ ,  $60-1^\circ$ ). B.p.  $306-5^\circ$  ( $304-5^\circ$ ,  $303^\circ$ ). Sol. EtOH, Et<sub>2</sub>O, hot ligroin. Spar. sol.  $H_2O$ .  $D_{20}^{25}$  1.0939. Heat of comb.  $C_p$  1327.3 Cal.

See first reference above.



**Naphthopicric Acid.**

See 2 : 4 : 5-Trinitro-1-naphthol.

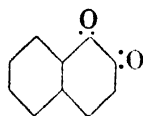
**Naphthopurpurin** (5 : 6 : 8-Trihydroxy-1 : 4-naphthoquinone) $C_{10}H_6O_5$ 

MW, 206

Red needles from  $C_6H_6$ . Sol. hot  $H_2O$ , EtOH, AcOH. Spar. sol. cold  $H_2O$ . NaOH sol. is purple-red. Conc.  $H_2SO_4 \rightarrow$  red sol. Colours wool orange-red in AcOH.

Jaubert, *Compt. rend.*, 1899, **129**, 684.Badische, D.R.P., 167,641, (*Chem. Zentr.*, 1906, I, 1126).**Naphthopyrogallol.**

See 1 : 2 : 3-Trihydroxynaphthalene.

**1 : 2-Naphthoquinone** ( $\beta$ -Naphthoquinone) $C_{10}H_6O_2$ 

MW, 158

Red needles from  $Et_2O$ , orange leaflets from  $C_6H_6$ . M.p.  $115-20^\circ$  decomp. Conc.  $H_2SO_4 \rightarrow$  green sol. Non-volatile in steam. Turns bluish-black on standing.  $SO_2 \rightarrow$  1 : 2-dihydroxynaphthalene.

Oxime : see Nitrosonaphthol.

Imide-oxime : see Nitrosonaphthylamine.

Phenylhydrazone : see Benzeneazonaphthol.

Tolylhydrazone : see Tolueneazonaphthol.

**1-o-Nitrophenylhydrazone**: 1-o-nitrobenzene-azo-2-naphthol.  $C_{16}H_{11}O_3N_3$ . MW, 293. Orange-red needles from AcOH. M.p.  $209^\circ$ . Spar. sol. EtOH. Insol. aq. alkalis. *Me ether* :  $C_{17}H_{13}O_3N_3$ . MW, 307. Red leaflets from EtOH. M.p.  $136-7^\circ$ . Sol.  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. EtOH,  $Et_2O$ , ligroin. Conc.  $H_2SO_4 \rightarrow$  red sol.  $B,2HNO_3$  : red cryst. M.p.  $103^\circ$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . *Et ether* :  $C_{18}H_{15}O_3N_3$ . MW, 321. Red plates from EtOH. M.p.  $111^\circ$ . Sol.  $C_6H_6$ ,  $CHCl_3$ , toluene. Spar. sol. EtOH,  $Et_2O$ . Conc.  $H_2SO_4 \rightarrow$  red sol.  $B,2HNO_3$  : red plates. M.p.  $105^\circ$  decomp.

**1-m-Nitrophenylhydrazone**: 1-m-nitrobenzene-azo-2-naphthol. Orange cryst. from toluene. M.p.  $193-4^\circ$ . Sol. alc. KOH with orange-red col. Insol. aq. alkalis. *Me ether* : red needles from EtOH. M.p.  $94-5^\circ$ . Sol.  $C_6H_6$ ,  $CHCl_3$ ,

Spar. sol. EtOH,  $Et_2O$ , ligroin.  $B,2HNO_3$  : golden-yellow leaflets. M.p.  $66-8^\circ$ . Sol. EtOH,  $CHCl_3$ . *Et ether* : red needles from EtOH. M.p.  $106-7^\circ$ . Sol.  $C_6H_6$ ,  $CHCl_3$ , toluene. Spar. sol. EtOH,  $Et_2O$ .  $B,2HNO_3$  : golden-yellow leaflets. M.p.  $70^\circ$ . *Acetyl* : red needles from EtOH. M.p.  $161-2^\circ$ . *Benzoyl* : orange-red needles from EtOH. M.p.  $171^\circ$ . Spar. sol. hot EtOH.

**1-p-Nitrophenylhydrazone**: 1-p-nitrobenzene-azo-2-naphthol. Paranitraniline Red, Para Red. Orange-brown plates from toluene. M.p.  $250-1^\circ$  ( $246^\circ$ ). Mod. sol. hot AcOH. Insol. EtOH. aq. alkalis. *Me ether* : red leaflets. M.p.  $128-9^\circ$ . Sol.  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. EtOH. Spar. sol.  $Et_2O$ .  $B,2HNO_3$  : green leaflets. M.p.  $75^\circ$  decomp. Sol.  $CHCl_3$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ , ligroin. *Et ether* : red leaflets from EtOH. M.p. about  $186^\circ$ . Sol.  $C_6H_6$ ,  $CHCl_3$ , toluene. Mod. sol. EtOH,  $Et_2O$ . Conc.  $H_2SO_4 \rightarrow$  red sol.  $B,2HNO_3$  : green leaflets. M.p.  $95-7^\circ$  decomp. *Acetyl* : orange needles from EtOH. M.p.  $192-3^\circ$ . Spar. sol. EtOH.

**2-o-Nitrophenylhydrazone**: 2-o-nitrobenzene-azo-1-naphthol. Brownish-red needles with green fluor. from isoamyl alcohol. M.p.  $218^\circ$ . Sol. hot xylene,  $Me_2CO$ . Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  green sol.

**2-p-Nitrophenylhydrazone**: 2-p-nitrobenzene-azo-1-naphthol. Deep red needles with green lustre. M.p.  $234-5^\circ$ . Sol. xylene, boiling isoamyl alcohol. Spar. sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Conc.  $H_2SO_4 \rightarrow$  red sol. *Acetyl* : red needles from AcOH. M.p.  $179.5^\circ$ .

**Dioxime**: yellow needles from EtOH. M.p.  $169^\circ$ . 1-*Me ether* : pale yellow cryst. M.p.  $161^\circ$ . 1-*Et ether* : greenish-yellow needles from EtOH. Aq. M.p.  $153^\circ$ . Mod. sol. EtOH,  $C_6H_6$ , AcOH. Insol.  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  red sol. 1-*Benzyl ether* : yellow prisms from  $Me_2CO-CHCl_3$ . M.p.  $168^\circ$ .

**Diphenylhydrazone**: yellow cryst. M.p.  $211-12^\circ$ .

**1-Semicarbazone**: golden-yellow leaflets from EtOH. Decomp. at  $184^\circ$ .

**2-Phenylsemicarbazone**: red needles from Py. M.p.  $250-1^\circ$ . Sol.  $CHCl_3$ , Py. Insol. other solvents.

**1-Anil**: green needles from EtOH. M.p.  $99-100^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

**1-Chloroimide**: yellow needles from  $C_6H_6$ -pet. ether. M.p.  $86-7^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. cold EtOH, AcOH, pet ether.

**2-Chloroimide**: brownish-yellow needles from  $C_6H_6$ -pet. ether. Decomp. at  $98^\circ$ .

*Dichloroimide*: yellow needles from  $C_6H_6$ -pet. ether. M.p. 105°.

Hantzsch, Glover, *Ber.*, 1906, **39**, 4171.

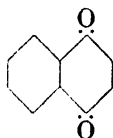
Lagodzinski, Hardine, *Ber.*, 1894, **27**, 3076.

Friedländer, Reinhardt, *Ber.*, 1894, **27**, 240.

Charrier, Ferreri, *gazz. chim. ital.*, 1913, **43**, ii, 236; i, 557; i, 239.

Bamberger, *Ber.*, 1897, **30**, 515; 1895, **28**, 849.

### 1 : 4-Naphthoquinone ( $\alpha$ -Naphthoquinone)



$C_{10}H_6O_2$

MW, 158

Yellow needles from EtOH or pet. ether. M.p. 125°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $CS_2$ . Mod. sol. EtOH, AcOH. Spar. sol.  $H_2O$ , pet. ether. Volatile in steam. Alkalis  $\rightarrow$  reddish-brown sol.

*Oxime*: see Nitrosonaphthol.

*Imide-oxime*: see Nitrosonaphthylamine.

*Phenylhydrazone*: see Benzeneazonaphthol.

*Tolylhydrazone*: see Tolueneazonaphthol.

*o*-Nitrophenylhydrazone: 4-*o*-nitrobenzeneazo-1-naphthol.  $C_{18}H_{11}O_3N_3$ . MW, 293. Dark red needles with bronze reflex from hot xylene. M.p. 244–5°. Sol. xylene, amyl alcohol. Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $Me_2CO$ ,  $CHCl_3$ .

*m*-Nitrophenylhydrazone: 4-*m*-nitrobenzeneazo-1-naphthol. Cryst. from EtOH. M.p. 288° decomp. *Et ether*: m.p. 145–6°. Sol. usual solvents.

*p*-Nitrophenylhydrazone: 4-*p*-nitrobenzeneazo-1-naphthol. Brown needles with blue reflex from  $PhNO_2$ . M.p. 277–9°. Spar. sol. most solvents. Reagent for magnesium. *Me ether*: red needles from EtOH. M.p. 169°. Sol.  $C_6H_6$ , AcOH. Less sol. EtOH. *Acetyl*: red needles from AcOH. M.p. 165–6°.

*Dioxime*: needles from EtOH.Aq. M.p. 207° decomp.

*Semicarbazone*: greenish-yellow cryst. from AcOH. M.p. 247° decomp. Insol.  $H_2O$ , EtOH.

*Chloroimide*: yellow needles from EtOH. M.p. 109–5°, decomp. at 130–3°. Sol. hot EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin.

*Dichloroimide*: yellow needles from EtOH or  $C_6H_6$ . M.p. 142–3°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

*Anil*: red cryst. from  $Et_2O$ . M.p. 103°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Dianil*: golden-yellow leaflets. M.p. 187°. Spar. sol. EtOH.

Jaeger, U.S.P., 1,692,126, (*Chem. Abstracts*, 1929, **23**, 612).

Conant, Freeman, *Organic Syntheses*, Collective Vol. I, 375.

Bamberger, Meimberg, *Ber.*, 1895, **28**, 1888.

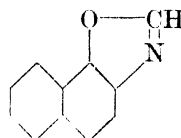
Meyer, Irschick, Schlösser, *Ber.*, 1914, **47**, 1749.

Bamberger, *Ber.*, 1895, **28**, 848.

### Naphthoresorcinol.

See 1 : 3-Dihydroxynaphthalene.

### $\alpha$ -Naphthoxazole



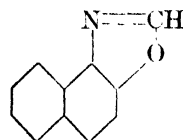
$C_{11}H_7ON$

MW, 169

Needles from ligroin. M.p. 79°. Sol. EtOH,  $Et_2O$ .

Fischer, *J. prakt. Chem.*, 1906, **73**, 440.

### $\beta$ -Naphthoxazole



$C_{11}H_7ON$

MW, 169

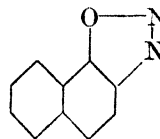
Leaflets from ligroin. M.p. 63.5–64°. Sol. EtOH,  $CHCl_3$ . Spar. sol. ligroin.

*B.HgCl\_2*: needles from EtOH. M.p. 183–4°.

*Picrate*: m.p. 133–4°.

See previous reference.

### $\alpha$ -Naphthoxdiazole



$C_{10}H_6ON_2$

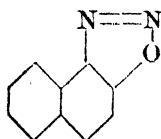
MW, 170

Yellow leaflets from pet. ether. M.p. 76°. Explodes at 112°. Sol. org. solvents.

Vesely, Dvořák, *Bull. soc. chim.*, 1923, **33**, 319.

Bamberger, *Ber.*, 1894, **27**, 680.

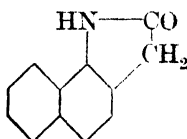
Geigy, D.R.P., 172,446, (*Chem. Zentr.*, 1906, II, 476).

**$\beta$ -Naphthoxdiazole** $C_{10}H_6ON_2$ 

MW, 170

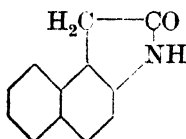
Yellow plates. M.p. 95°.

See last two references above.

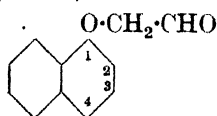
 **$\alpha$ -Naphthoxindole** $C_{12}H_9ON$ 

MW, 183

Needles from EtOH.Aq. or AcOH. M.p. 247°.

Hinsberg, *Ber.*, 1888, 21, 116.Mayer, Oppenheimer, *Ber.*, 1918, 51, 1245. **$\beta$ -Naphthoxindole** $C_{12}H_9ON$ 

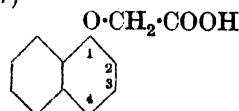
MW, 183

Pale greenish needles from EtOH. M.p. 234°. Mod. sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. H<sub>2</sub>O.Hinsberg, *Ber.*, 1888, 21, 114.M.L.B., D.R.P., 216,639, (*Chem. Zentr.*, 1910, I, 130). **$\alpha$ -Naphthoxyacetaldehyde (Glycollic aldehyde 1-naphthyl ether)** $C_{12}H_{10}O_2$ 

MW, 186

*Hydrate*:  $C_{10}H_7 \cdot O \cdot CH_2 \cdot CH(OH)_2$ . MW, 204. Cryst. from H<sub>2</sub>O. M.p. 86°.*Di-Et acetal*: b.p. 207-8°/18 mm.  $D^{14}_4$  1.0698.  $n^{18}_D$  1.5610.  $ZnCl_2$  in hot AcOH  $\rightarrow$   $\alpha$ -naphthafuran.*Oxime*: m.p. 108°.*Semicarbazone*: m.p. 149-50°.Stoermer, *Ber.*, 1897, 30, 1703. **$\beta$ -Naphthoxyacetaldehyde (Glycollic aldehyde 2-naphthyl ether).***Hydrate*: needles from H<sub>2</sub>O. M.p. 87°. Spar. sol. H<sub>2</sub>O. Reduces Fehling's and  $NH_3 \cdot AgNO_3$ .*Di-Et acetal*: yellow oil. B.p. 240°/60 mm., 206-7°/17 mm.  $D^{14}_4$  1.0654.  $n^{18}_D$  1.557. Very spar. volatile in steam.  $ZnCl_2$  in hot AcOH  $\rightarrow$   $\beta$ -naphthafuran.*Oxime*: cryst. M.p. 123.5°.*Semicarbazone*: white cryst. with blue fluor. M.p. 182°.*Phenylhydrazone*: cryst. M.p. 145°. Turns brown in air.

See previous reference and also

Hesse, *Ber.*, 1897, 30, 1439. **$\alpha$ -Naphthoxyacetic Acid (Glycollic acid 1-naphthyl ether)** $C_{12}H_{10}O_3$ 

MW, 202

Prisms. M.p. 190°. Very sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. $NH_4$  salt: needles. M.p. 119-20°.*Ester*:  $C_{14}H_{14}O_3$ . MW, 230. Cryst. M.p. 173-4°. Sol. EtOH, Et<sub>2</sub>O.*Amide*:  $C_{12}H_{11}O_2N$ . MW, 201. Leaflets or needles from EtOH.Aq. M.p. 155°. Sol. EtOH. Spar. sol. H<sub>2</sub>O.*Chloride*:  $C_{12}H_9O_2Cl$ . MW, 220.5. Oil. B.p. 194°/10 mm.*Anilide*: needles from EtOH. M.p. 144°.*p-Phenetidine*: needles. M.p. 145-6°. Mod. sol. EtOH.Spica, *Gazz. chim. ital.*, 1886, 16, 438.Ingham, Stephen, Timpe, *J. Chem. Soc.*, 1931, 897. **$\beta$ -Naphthoxyacetic Acid (Glycollic acid 2-naphthyl ether).**Prisms from H<sub>2</sub>O. M.p. 156°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Mod. sol. hot H<sub>2</sub>O. $NH_4$  salt: cryst. M.p. 180°. Spar. sol. cold H<sub>2</sub>O.*Ester*: plates. M.p. 48-9°. Triboluminescent.*m-Tolyl ester*:  $C_{19}H_{16}O_3$ . MW, 292. Needles from pet. ether. M.p. 91-2°.*Amide*: needles or plates. M.p. 147°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.*Chloride*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 54°. B.p. 207°/10 mm.*Nitrile*:  $C_{12}H_9ON$ . MW, 183. Leaflets. M.p. 72°. Sol. EtOH, Et<sub>2</sub>O.*Anilide*: needles from EtOH. M.p. 145°.

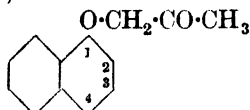
*p*-Phenetidide: needles. M.p. 164 5°. Spar. sol. hot EtOH.

See previous references and also  
Lees, Shedden, *J. Chem. Soc.*, 1903, 83, 758.

Spitzer, *Ber.*, 1901, **34**, 3192.

Stoermer, *Ber.*, 1897, **30**, 1702.

**$\alpha$ -Naphthoxyacetone** (*Hydroxyacetone 1-naphthyl ether*)



$C_{13}H_{12}O_2$

MW, 200

Liq. B.p. 205–8°/14 mm.

Semicarbazone: m.p. 103°.

Stoermer, *Ann.*, 1900, **312**, 313.

**$\beta$ -Naphthoxyacetone** (*Hydroxyacetone 2-naphthyl ether*).

Plates. M.p. 78°. Spar. volatile in steam.

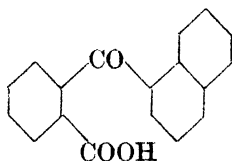
Oxime: leaflets. M.p. 123°.

Semicarbazone: m.p. 203°.

Phenylhydrazone: leaflets. M.p. 154°.

See previous reference.

***o*-1-Naphthoylbenzoic Acid**



$C_{18}H_{12}O_3$

MW, 276

Prisms from EtOH.Aq. M.p. 176–4°. Sol. 12 parts  $CHCl_3$ . Insol.  $H_2O$ . Sol. conc.  $H_2SO_4$  with violet col. Heat at 275°  $\rightarrow$  naphthanthraquinone. KOH fusion  $\rightarrow$  1-naphthoic and benzoic acids.

*Me ester*:  $C_{19}H_{14}O_3$ . MW, 290. Yellow cryst. from MeOH. M.p. 120°. Conc.  $H_2SO_4$   $\rightarrow$  yellow sol.  $\rightarrow$  red on standing.

*Et ester*:  $C_{20}H_{16}O_3$ . MW, 304. M.p. 123–4°.

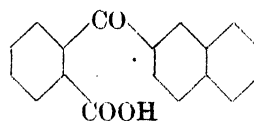
*Amide*:  $C_{18}H_{13}O_2N$ . MW, 275. Needles from. EtOH. M.p. 215°. Sol.  $C_6H_6$ . Spar. sol.  $H_2O$ , EtOH.

Heller, Schulke, *Ber.*, 1908, **41**, 3633.

Graebe, *Ann.*, 1889, **340**, 251.

Groggins, Newton, *Ind. Eng. Chem.*, 1930, **22**, 157.

***o*-2-Naphthoylbenzoic Acid**



$C_{18}H_{12}O_3$

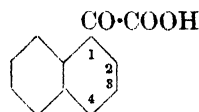
MW, 276

Needles from toluene. M.p. 165°.

*Acetyl*: cryst. from  $Ac_2O$ . M.p. 140°. Sol.  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ ,  $CHCl_3$ . Insol. EtOH.

McMullen, *J. Am. Chem. Soc.*, 1922, **44**, 2058.

**1-Naphthoylformic Acid** (*1-Naphthylglyoxylic acid*)



$C_{12}H_8O_3$

MW, 200

Needles. M.p. 107–8° decomp. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $CS_2$ , pet. ether.  $NaHg \rightarrow$  1-naphthylglycollic acid.  $KMnO_4 \rightarrow$  1-naphthoic acid.

*Et ester*:  $C_{14}H_{12}O_3$ . MW, 228. Liq. B.p. 213–15°/23 mm.  $D^{20}_D$  1.19. *Picrate*: yellow needles. M.p. 77°.

*Amide*:  $C_{12}H_9O_2N$ . MW, 199. Needles from EtOH. M.p. 151°.

*Nitrile*:  $C_{12}H_7ON$ . MW, 181. Yellow needles from  $Et_2O$ . M.p. 101°. B.p. 230°/85 mm. Decomp. by boiling  $H_2O$ .

*Oxime*: m.p. 193–5°.

Rousset, *Bull. soc. chim.*, 1897, **17**, 301.

Boessneck, *Ber.*, 1882, **15**, 3065; 1883, **16**, 640.

**2-Naphthoylformic Acid** (*2-Naphthylglyoxylic acid*).

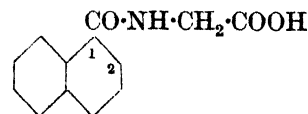
Cryst. from  $C_6H_6$ . M.p. 171°. Sol.  $H_2O$ .

*Semicarbazone*: cryst. from EtOH. M.p. 230°.

*Thiosemicarbazone*: yellowish cryst. from EtOH. M.p. 226°. Spar. sol. other solvents.

Popovici, *Compt. rend.*, 1930, **191**, 210.

**1-N-Naphthoylglycine** ( *$\alpha$ -Naphthoylaminoacetic acid*)



$C_{13}H_{11}O_3N$

MW, 229

Needles from  $H_2O$ . M.p. 153°.

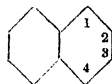
Cohn, *Ber.*, 1894, **27**, 2911.

**2-N-Naphthoylglycine** ( $\beta$ -Naphthoylaminoacetic acid).

Needles from  $\text{H}_2\text{O}$ . M.p. 169–70°. Sol. MeOH, EtOH,  $\text{Me}_2\text{CO}$ , AcOEt. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ ,  $\text{CHCl}_3$ , pet. ether.

Friedmann, Türk, *Biochem. Z.*, 1913, **55**, 466.

### 1-Naphthoylhydrazine



$\text{C}_{11}\text{H}_{10}\text{ON}_2$

MW, 186

Needles from EtOH. M.p. 166°.

Stollé, Zinsser, *J. prakt. Chem.*, 1906, **74**, 19.

### 2-Naphthoylhydrazine.

Needles from 50% EtOH. M.p. 147.5°. Sol. boiling  $\text{H}_2\text{O}$ , EtOH,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , AcOH. Sol. HCl. Spar. sol. AcOH.Aq. Reduces  $\text{NH}_3\cdot\text{AgNO}_3$ .

*N*-Acetyl: cryst. from 50% EtOH. M.p. 138°. Sol. boiling  $\text{H}_2\text{O}$ , EtOH,  $\text{CHCl}_3$ , AcOH.

*N*'-Naphthoyl: plates from AcOH. M.p. 241°. Spar. sol. usual solvents.

Goldstein, Cornamusaz, *Helv. Chim. Acta*, 1932, **15**, 941.

**1-N-Naphthoylhydroxylamine** ( $\alpha$ -Naphthohydroxamic acid)



$\text{C}_{11}\text{H}_9\text{O}_2\text{N}$

MW, 187

Plates from EtOH. M.p. 186–7° decomp. Sol. hot EtOH. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Boiling aq. sol. gives deep wine-red col. with  $\text{FeCl}_3$ .

O-1-Naphthoyl: needles from EtOH, prisms from  $\text{C}_6\text{H}_6$ . M.p. 150°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . No col. with  $\text{FeCl}_3$ .

Ekstrand, *Ber.*, 1887, **20**, 1355.

**2-N-Naphthoylhydroxylamine** ( $\beta$ -Naphthohydroxamic acid).

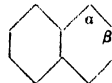
Plates from EtOH. M.p. 168°. Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .  $\text{FeCl}_3 \rightarrow$  wine-red col.

O-1-Naphthoyl: needles from EtOH. M.p. 160°. Mod. sol. EtOH.

O-2-Naphthoyl: needles from EtOH. M.p. 171°. Spar. sol. EtOH.

See previous reference.

### 2- $\alpha$ -Naphthoylpropionic Acid



$\text{C}_{14}\text{H}_{12}\text{O}_3$

MW, 228

Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 131–2° (118°).

Giua, *Ber.*, 1914, **47**, 2115.

Schroeter, Mullen, Huang, *Ber.*, 1929, **62**, 656.

### 2- $\beta$ -Naphthoylpropionic Acid.

Needles from EtOH.Aq. M.p. 174° (165°). Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . KOH fusion  $\rightarrow$  2-naphthoic acid + naphthalene.

*Me ester*:  $\text{C}_{15}\text{H}_{14}\text{O}_3$ . MW, 242. Prisms. M.p. 74°. Very sol. most org. solvents. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

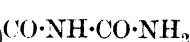
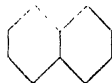
*Et ester*:  $\text{C}_{16}\text{H}_{16}\text{O}_3$ . MW, 256. Cryst. from EtOH. M.p. 47–8°.

*Oxime*: cryst. from EtOH.Aq. M.p. 135–6°.

See previous references and also

Borsche, Sauernheimer, *Ber.*, 1914, **47**, 1647.

### N-2-Naphthoylurea



$\text{C}_{12}\text{H}_{10}\text{O}_2\text{N}_2$

MW, 214

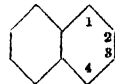
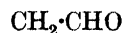
Needles from EtOH. M.p. 215°. Mod. sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ .

Vieth, *Ann.*, 1876, **180**, 322.

### Naphthhydroxamic Acid.

See N-Naphthoylhydroxylamine.

### 1-Naphthylacetaldehyde



$\text{C}_{12}\text{H}_{10}\text{O}$

MW, 170

Liq. B.p. 163–6°/13 mm. Alk.  $\text{Ag}_2\text{O} \rightarrow$  1-naphthylacetic acid. Forms bisulphite comp.

*Semicarbazone*: m.p. 208°.

*Oxime*: needles from  $\text{CCl}_4$ . M.p. 118°.

Tiffeneau, Daudel, *Compt. rend.*, 1908, **147**, 679.

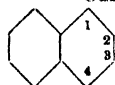
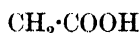
### 2-Naphthylacetaldehyde.

*Oxime*: needles from  $\text{CCl}_4$ . M.p. 120°.

Mayer, Sieglitz, *Ber.*, 1922, **55**, 1858.

## 1-Naphthylacetic Acid

### 1-Naphthylacetic Acid



$\text{C}_{12}\text{H}_{10}\text{O}_2$

MW, 186

Needles from  $\text{H}_2\text{O}$ . M.p.  $131^\circ$  ( $129^\circ$ ). Sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ , AcOH,  $\text{C}_6\text{H}_6$ . Heat with lime  $\rightarrow$  1-methylnaphthalene.

*Et ester*:  $\text{C}_{14}\text{H}_{14}\text{O}_2$ . MW, 214. Oil. B.p.  $177-9^\circ/13$  mm.

*Chloride*:  $\text{C}_{12}\text{H}_9\text{OCl}$ . MW, 204.5. Yellowish oil. B.p.  $188^\circ/23$  mm.,  $148-55^\circ/0.05$  mm.

*Amide*:  $\text{C}_{12}\text{H}_{11}\text{ON}$ . MW, 185. Needles from EtOH. M.p.  $180-1^\circ$  ( $154^\circ$ ). Sol. hot  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ , AcOH. Difficult to hydrolyse.

*Nitrile*:  $\text{C}_{12}\text{H}_9\text{N}$ . MW, 167. Oil. F.p.  $5^\circ$ . B.p.  $183-7^\circ/13$  mm.

Mayer, Oppenheimer, *Ber.*, 1916, **49**, 2139.  
Wislicenus, Elvert, *ibid.*, 2822.

Weitzenböck, Lieb, *Monatsh.*, 1912, **33**, 556.

Willegerodt, Scholz, *J. prakt. Chem.*, 1910, **81**, 387.

Keach, *J. Am. Chem. Soc.*, 1933, **55**, 2974.

I.G., D.R.P., 562,391, (*Chem. Abstracts*, 1933, **27**, 734).

Gen. Aniline Works, U.S.P., 1,951,686, (*Chem. Abstracts*, 1934, **28**, 3423).

### 2-Naphthylacetic Acid.

Leaflets from  $\text{H}_2\text{O}$ , cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $142^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , AcOEt, ligroin. Dist.  $\rightarrow$  2-methylnaphthalene.

*Et ester*: cryst. from pet. ether. M.p.  $31-2^\circ$ . B.p.  $186-7^\circ/14$  mm.

*Amide*: leaflets from  $\text{H}_2\text{O}$ . M.p.  $200^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Less sol. hot  $\text{H}_2\text{O}$ .

*Nitrile*: needles and leaflets from 50% EtOH. M.p.  $86^\circ$  ( $79-81^\circ$ ). Very sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ , AcOEt. Spar. sol. EtOH, warm  $\text{H}_2\text{O}$ . Insol. pet. ether.

Kikkaji, *Biochem. Z.*, 1911, **35**, 77.

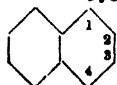
Wislicenus, Elvert, *Ber.*, 1916, **49**, 2828.

Willegerodt, *J. prakt. Chem.*, 1909, **80**, 188.

Blank, *Ber.*, 1896, **29**, 2374.

Mayer, Oppenheimer, *Ber.*, 1916, **49**, 2140.

### 1-Naphthylacetylene



$\text{C}_{12}\text{H}_8$

MW, 152

## 2-β-Naphthylacrylic Acid

Liq. B.p.  $143-4^\circ/25$  mm.  $\text{H}_2\text{SO}_4 \rightarrow$  methyl 1-naphthyl ketone.

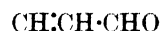
Leroy, *Bull. soc. chim.*, 1891, **6**, 386.

### 2-Naphthylacetylene.

Cryst. M.p.  $36^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ .  $\text{H}_2\text{SO}_4 \rightarrow$  methyl 2-naphthyl ketone.

Leroy, *Bull. soc. chim.*, 1892, **7**, 648.

### 2-α-Naphthylacrolein



$\text{C}_{13}\text{H}_{10}\text{O}$

MW, 182

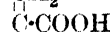
Pale yellow needles from pet. ether. M.p.  $48^\circ$ . B.p.  $160-70^\circ/0.4$  mm.

*Oxime*: m.p.  $152^\circ$ .

*Semicarbazone*: light yellow. M.p.  $228^\circ$ .

v. Braun, Nelles, *Ber.*, 1933, **66**, 1470.

### 1-α-Naphthylacrylic Acid



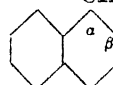
$\text{C}_{13}\text{H}_{10}\text{O}_2$

MW, 198

Plates from  $\text{C}_6\text{H}_6$ . M.p.  $152-4^\circ$ . Sol.  $\text{Et}_2\text{O}$ . Mod. sol. warm  $\text{C}_6\text{H}_6$ , hot  $\text{H}_2\text{O}$ . Spar. sol. pet. ether.

Wislicenus, Butterfass, Koken, *Ann.*, 1924, **436**, 77.

### 2-α-Naphthylacrylic Acid



$\text{C}_{13}\text{H}_{10}\text{O}_2$

MW, 198

Needles from EtOH. M.p.  $211-12^\circ$  ( $205^\circ$ ). Sol. 2000 parts boiling  $\text{H}_2\text{O}$ . Sol.  $\text{Et}_2\text{O}$ .  $\text{KMnO}_4 \rightarrow$  1-naphthaldehyde.

*Chloride*:  $\text{C}_{13}\text{H}_9\text{OCl}$ . MW, 216.5. Liq. B.p.  $180^\circ/0.4$  mm.

*Anilide*: cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $212^\circ$ .

Brandis, *Ber.*, 1889, **22**, 2153.

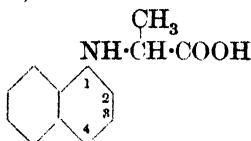
v. Braun, Nelles, *Ber.*, 1933, **66**, 1470.

West, *J. Am. Chem. Soc.*, 1920, **42**, 1664.

### 2-β-Naphthylacrylic Acid.

Needles. M.p.  $203^\circ$  ( $196^\circ$ ). Mod. sol. hot  $\text{H}_2\text{O}$ .

Monier-Williams, *J. Chem. Soc.*, 1906, **89**, 277.

**N-1-Naphthylalanine** (1- $\alpha$ -Naphthylamino-propionic acid) $C_{13}H_{13}O_2N$ 

MW, 215

Cryst. from  $C_6H_6$ . M.p. 161°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , dil. HCl,  $Na_2CO_3$ .

*Et ester*:  $C_{15}H_{17}O_2N$ . MW, 243. (Cryst. from EtOH. M.p. 65.5°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $Me_2CO$ , AcOH,  $CS_2$ . Spar. sol. cold EtOH, ligroin.

*Nitrile*:  $C_{13}H_{12}N_2$ . MW, 196. Prisms from EtOH.Aq. M.p. 104-5°. Sol.  $C_6H_6$ , toluene. Mod. sol. EtOH.

Bischoff, Hausdörfer, *Ber.*, 1892, 25, 2309.  
Maron, D.R.P., 144,536, (*Chem. Zentr.*, 1903, II, 779).

**N-2-Naphthylalanine** (1- $\beta$ -Naphthylamino-propionic acid).

Cryst. from EtOH. M.p. 170-1°. Spar. sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ . Insol.  $CS_2$ , ligroin.

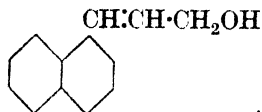
*Et ester*: prisms from EtOH. M.p. 84°. Sol. most org. solvents.

*N-Acetyl*: prisms from  $CHCl_3$ -ligroin. M.p. 199-200°. Sol. EtOH,  $Me_2CO$ , AcOH,  $CHCl_3$ . Spar. sol. cold  $C_6H_6$ . Insol.  $CS_2$ , ligroin.

Bischoff, Hausdörfer, *Ber.*, 1892, 25, 2311.

**1- $\alpha$ -Naphthylallyl Alcohol.**

See Vinyl-1-naphthylcarbinol.

**3- $\alpha$ -Naphthylallyl Alcohol** (1- $\gamma$ -Hydroxy-propenylnaphthalene) $C_{13}H_{12}O$ 

MW, 184

Cryst. M.p. 39-40°. B.p. 209-10°/18 mm.

*Acetyl*: b.p. 210-11°/9 mm.

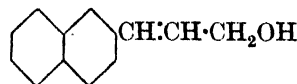
*p-Nitrobenzoyl*: needles from AcOH. M.p. 138-9°.

*Phenylurethane*: needles from  $C_6H_6$ -pet. ether. M.p. 120°.

Burton, *J. Chem. Soc.*, 1931, 761.

**1- $\beta$ -Naphthylallyl Alcohol.**

See Vinyl-2-naphthylcarbinol.

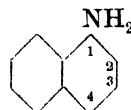
**3- $\beta$ -Naphthylallyl Alcohol** (2- $\gamma$ -Hydroxy-propenylnaphthalene) $C_{13}H_{12}O$ 

MW, 184

Needles from  $C_6H_6$ . M.p. 116°.

*p-Nitrobenzoyl*: plates from EtOH. M.p. 148°. Spar. sol. EtOH.

Burton, *J. Chem. Soc.*, 1931, 761.

**1-Naphthylamine** ( $\alpha$ -Naphthylamine) $C_{10}H_9N$ 

MW, 143

Needles from EtOH.Aq. or  $Et_2O$ . M.p. 50°. B.p. 300-8°/760 mm. Very sol. EtOH,  $Et_2O$ . Sol. to 0.167% in  $H_2O$  at 25°.  $D_{25}^{25}$  1.1229,  $D_{25}^{50}$  1.1144.  $n_D^{25}$  1.67034. Heat of comb.  $C_p$  1268 Cal.  $k = 9.9 \times 10^{-11}$  at 25°. Sublimes. Bitter taste. Volatile in steam. Reduces warm  $NH_3$ .  $AgNO_3$ .

*Chloroacetic acid salt*: m.p. 93-4°.

*m-Nitrobenzoic acid salt*: needles from  $C_6H_6$ . M.p. 105-6°.

*2:4:6-Trinitrobenzoic acid salt*: needles. M.p. 197°.

*Benzenesulphonic acid salt*: needles. M.p. 225°.

*p-Toluenesulphonic acid salt*: prisms. M.p. 239°.

$C_{10}H_9N, C_6H_3(NO_2)_3$ -1:3:5: red prisms. M.p. 214°. Sol. EtOH, AcOH. Spar. sol. ligroin.

*Picrate*: greenish-yellow prisms from EtOH. M.p. 181-2° decomp.

*Styphnate*: cryst. from EtOH. M.p. 181-2°.

*N-Me*: see *N-Methyl-1-naphthylamine*.

*N-Di-Me*: see *Dimethyl-1-naphthylamine*.

*N-Et*: see *N-Ethyl-1-naphthylamine*.

*N-Di-Et*: see *Diethyl-1-naphthylamine*.

*N-Propyl*:  $C_{13}H_{15}N$ . MW, 185. Yellow oil. B.p. 316-18°/771 mm. *N-Acetyl*: leaflets from EtOH.Aq. M.p. 94°.

*N-Dipropyl*:  $C_{19}H_{21}N$ . MW, 227. Oil. B.p. above 300°.  $B_2H_2PtCl_6$ : decomp. at 212°.

*N-Pentadecyl*: needles from EtOH. M.p. 47-8°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $Me_2CO$ . *B, HCl*: needles from pet. ether. M.p. 92-4°. Sol.  $CHCl_3$ . Spar. sol.  $Et_2O$ .

*N-Heptadecyl*: cryst. from EtOH. M.p. 53-5°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , pet. ether. Insol.  $H_2O$ . Sol. conc.  $H_2SO_4$ . *B, HCl*: plates from pet.

ether. M.p. 96–7°. Sol.  $\text{CHCl}_3$ . Insol.  $\text{Me}_2\text{CO}$ ,  $\text{Et}_2\text{O}$ , cold pet. ether.

*N-Phenyl*: see *N-Phenyl-1-naphthylamine*.

*N-m-Chlorophenyl*: cryst. from EtOH. M.p. 73°. B.p. 238–41°/12 mm. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*N-p-Chlorophenyl*: yellowish prisms from EtOH.Aq. M.p. 102–3°. Conc.  $\text{H}_2\text{SO}_4 + \text{HNO}_2 \rightarrow$  blood-red col.

*N-2:4-Dinitrophenyl*: orange-red needles from EtOH. M.p. 190–5°. Very sol.  $\text{C}_6\text{H}_6$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Spar. sol. EtOH, AcOH. Insol.  $\text{H}_2\text{O}$ .

*N-2:4:6-Trinitrophenyl*: see *N-Picryl-1-naphthylamine*.

*N-Diphenyl*: see *Diphenyl-1-naphthylamine*.

*N-o-Tolyl*: needles from ligroin. M.p. 94–5°. B.p. 198–202°/9 mm. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*N-m-Tolyl*: solid. B.p. 234–5°/11 mm.

*N-p-Tolyl*: prisms from EtOH. M.p. 79°. B.p. 230°/10 mm. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold EtOH. Sols. show blue fluor. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. turning blue and then yellowish-brown on adding  $\text{HNO}_2$ . *Acetyl*: cryst. from AcOEt. M.p. 124°. Sol. usual solvents.

*N-Benzyl*: see *N-Benzyl-1-naphthylamine*.

*N-o-Nitrobenzyl*: golden-yellow needles from EtOH. M.p. 97°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , AcOH. Spar. sol. cold EtOH, ligroin. *Acetyl*: leaflets from EtOH. M.p. 130°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*N-m-Nitrobenzyl*: orange-yellow prisms from EtOH. M.p. 94°. Mod. sol.  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH,  $\text{Et}_2\text{O}$ , ligroin.

*N-p-Nitrobenzyl*: orange-yellow leaflets from EtOH. M.p. 126–7°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, ligroin. *Acetyl*: needles from EtOH. M.p. 112–13°.

*N-Dibenzyl*: see *Dibenzyl-1-naphthylamine*.

*N-Naphthyl*: see *Dinaphthylamine*.

*N-Benzylidene*: see *Benzylidene-1-naphthylamine*.

*N-o-Nitrobenzylidene*: yellow. M.p. 118°.

*N-m-Nitrobenzylidene*: yellow plates from ligroin. M.p. 102–3°.

*N-Cinnamylidene*: plates and needles from EtOH. M.p. 65°.

*N-Salicylidene*: orange prisms from MeOH. M.p. 45–5°.

*N-Anisylidene*: plates from EtOH. M.p. 100–1°.

*Formyl*: needles from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 138–5°. Sol. boiling  $\text{H}_2\text{O}$ .

*Acetyl*: 1-acetnaphthalide. Cryst. from EtOH. M.p. 160°. Very sol. EtOH. Mod. sol. boiling  $\text{H}_2\text{O}$ .  $\text{B}, \text{C}_6\text{H}_3(\text{NO}_2)_3$ -1:3:5: yellow needles. M.p. 140–5°.

*Chloroacetyl*: needles from EtOH. M.p. 161°.

*Propionyl*: cryst. from EtOH. M.p. 116°.

*Butyryl*: cryst. from EtOH. M.p. 120°.

*Valeryl*: cryst. from EtOH. M.p. 111°.

*Caproyl*: m.p. 112°.

*Heptylyl*: m.p. 106°.

*Caprylyl*: m.p. 95°.

*Pelargonyl*: m.p. 91°.

*Lauryl*: m.p. 100°.

*Myristyl*: needles. M.p. 110° (105°). B.p. 162–5°/10 mm.

*Palmityl*: needles. M.p. 112–8°. B.p. 182°/10 mm.

*Stearyl*: needles. M.p. 110–8°. B.p. 205°/10 mm.

*Benzoyl*: 1-benzonaphthalide. Needles from EtOH or AcOH. M.p. 161–2°. Sol. AcOH, EtOH.Aq. Spar. sol. EtOH.  $\text{B}, \text{C}_6\text{H}_3(\text{NO}_2)_3$ -1:3:5: yellow needles from  $\text{CCl}_4$ . M.p. 131–2°. *Benzene-sulphonyl*: cryst. from  $\text{C}_6\text{H}_6$ -ligroin. M.p. 193–4°.

*1-Naphthoyl*: cryst. M.p. 244°. Spar. sol. EtOH. Insol.  $\text{C}_6\text{H}_6$ .

*2-Naphthoyl*: needles or prisms from EtOH. M.p. 160°. Mod. sol. EtOH,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ .

*Benzenesulphonyl*: needles from EtOH. M.p. 168–9°.

*p-Toluenesulphonyl*: prisms from EtOH. M.p. 147°.

Knoll, D.R.P., 241,853, (*Chem. Zentr.*, 1912, I, 178).

Knoevenagel, *J. prakt. Chem.*, 1914, **89**, 17, 22.

Liebermann, Jacobsen, *Ann.*, 1882, **211**, 42.

Mailhe, Murat, *Bull. soc. chim.*, 1910, **7**, 955.

Dehn, *J. Am. Chem. Soc.*, 1912, **34**, 1405.

De'Conno, *Gazz. chim. ital.*, 1917, **47**, i, 98, 122.

Robertson, *J. Chem. Soc.*, 1908, **93**, 1037.

Jacobs, Heidelberger, *J. Am. Chem. Soc.*, 1917, **39**, 1446.

Le Sueur, *J. Chem. Soc.*, 1911, **99**, 831.

West, *J. Chem. Soc.*, 1925, **127**, 494.

Popov, *Chem. Abstracts*, 1934, **28**, 1671.

## 2-Naphthylamine ( $\beta$ -Naphthylamine).

Leaflets from  $\text{H}_2\text{O}$ . M.p. 113°. B.p. 294°. Sol.  $\text{H}_2\text{O}$ .  $D_4^{20}$  1.0614. Heat of comb.  $\text{C}_p$  1267.5 Cal.  $n_D^{20}$  1.64927.  $k = 2.0 \times 10^{-10}$  at 25°. Volatile in steam. Heat at 230° with trace of I  $\rightarrow$  2:2'-dinaphthylamine. S + I at 230°  $\rightarrow$  thio-2:2'-dinaphthylamine. Reduces warm  $\text{NH}_3$ .  $\text{AgNO}_3$ .



*m*-Nitrobenzoic acid salt : pale yellow needles. M.p. 119°.

2 : 4 : 6-Trinitrobenzoic acid salt : m.p. 156°.

$C_{10}H_9N, C_6H_3(NO_2)_3 \cdot 1 : 3 : 5$  : red needles. M.p. 162°. Sol. most org. solvents.

*Picrate* : yellow needles from  $H_2O$ . M.p. 195° decomp.

*Styphate* : cryst. from EtOH. M.p. 194–5°.

*B, HCl* : leaflets. M.p. 254°.

*N-Me* : see *N*-Methyl-2-naphthylamine.

*N-Di-Me* : see Dimethyl-2-naphthylamine.

*N-Et* : see *N*-Ethyl-2-naphthylamine.

*N-Di-Et* : see Diethyl-2-naphthylamine.

*N-Propyl* : oil. B.p. 322–4°/761 mm.

*N-Butyl* : oil with characteristic odour. B.p. 348–50°.  $D_4^{20}$  1.02. Insol.  $H_2O$ . Shows faint violet fluor. *B, HCl* : m.p. 176–8°. *p*-Bromobenzoil : prisms from EtOH. M.p. 125°.

*N-Pentadecyl* : plates from EtOH. M.p. 53–4°. Sol.  $Et_2O$ ,  $C_6H_6$ , pet. ether. Insol.  $H_2O$ . Sol. conc.  $H_2SO_4$ . *B, HCl* : needles from  $CHCl_3$ -pet. ether. M.p. 176–7°. Sol. hot  $CHCl_3$ . Insol. EtOH,  $Et_2O$ , pet. ether.

*N-Heptadecyl* : plates from EtOH. M.p. 59–61°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol.  $Me_2CO$ , pet. ether. Insol.  $H_2O$ . Sol. conc.  $H_2SO_4$ . *B, HCl* : needles from  $CHCl_3$ -pet. ether. M.p. 170–1°. Sol. boiling  $CHCl_3$ . Insol. EtOH,  $Et_2O$ , pet. ether.

*N-Phenyl* : see *N*-Phenyl-2-naphthylamine.

*N-o-Chlorophenyl* : cryst. from EtOH. M.p. 89°. B.p. 236–8°/13.5 mm. Conc.  $H_2SO_4$  → yellow sol. → red on adding  $HNO_3$ .

*N-m-Chlorophenyl* : cryst. from EtOH. M.p. 101°. B.p. 250–3°/11 mm. Conc.  $H_2SO_4$  → yellow sol. → brown on adding  $HNO_3$ .

*N-p-Chlorophenyl* : cryst. from EtOH. M.p. 101°. B.p. 251.5°/13 mm. Conc.  $H_2SO_4$  → yellowish sol. → orange-red on adding  $HNO_3$ .

*N-o-Nitrophenyl* : orange needles from EtOH. M.p. 110°.

*N-p-Nitrophenyl* : yellow cryst. from  $C_6H_6$ . M.p. 283–4°. Spar. sol. cold EtOH,  $Et_2O$ . Sol.  $C_6H_6$ .

*N-2 : 4-Dinitrophenyl* : red cryst. M.p. 183° (169°). Sol. hot AcOH,  $Me_2CO$ . Mod. sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. EtOH.

*N-2 : 4 : 6-Trinitrophenyl* : see *N*-Picryl-2-naphthylamine.

*N-o-Tolyl* : cryst. from ligroin. M.p. 95° (105°). B.p. 400–5°, 235–7°/14 mm. Sol.  $C_6H_6$ , EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ , ligroin. *Benzoyl* : leaflets from EtOH. M.p. 117–18°.

*N-m-Tolyl* : cryst. from EtOH. M.p. 68–9°. B.p. 243–6°/15 mm. Sol. hot EtOH with blue fluor.,  $C_6H_6$ ,  $Et_2O$ ,  $Me_2CO$ , ligroin.

*N-p-Tolyl* : leaflets from hot EtOH. M.p. 102–3°. Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. cold EtOH, ligroin. Sols. show blue fluor. *Acetyl* : needles. M.p. 85°. Mod. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . *Benzoyl* : needles from EtOH. M.p. 139°.

*N-Benzyl* : see *N*-Benzyl-2-naphthylamine.

*N-o-Nitrobenzyl* : red leaflets from EtOH or AcOH. M.p. 162°. Sol.  $C_6H_6$ ,  $CHCl_3$ ,  $CS_2$ . Spar. other solvents. *Acetyl* : prisms from EtOH. M.p. 117–18°.

*N-m-Nitrobenzyl* : yellow needles from EtOH.Aq. M.p. 80°.

*N-p-Nitrobenzyl* : yellow leaflets from EtOH.Aq. M.p. 121.5°. Sol.  $C_6H_6$ ,  $Et_2O$ . Spar. sol. cold EtOH.

*N-Naphthyl* : see Dinaphthylamine.

*N-Benzylidene* : see Benzylidene-2-naphthylamine.

*N-o-Nitrobenzylidene* : yellow leaflets from EtOH. M.p. 91°. Sol. org. most solvents.

*N-m-Nitrobenzylidene* : yellow plates from EtOH. M.p. 90°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. EtOH.

*N-p-Nitrobenzylidene* : golden-yellow needles. M.p. 120–1°. Very sol.  $Et_2O$ ,  $Me_2CO$ .

*N-Cinnamylidene* : yellow cryst. from EtOH. M.p. 125°. Very sol.  $CHCl_3$ . Sol. MeOH,  $C_6H_6$ ,  $Et_2O$ . Mod. sol.  $Me_2CO$ , AcOEt, toluene.

*N-Salicylidene* : reddish-yellow needles from EtOH. M.p. 126°.

*N-Furfurylidene* : leaflets from EtOH. M.p. 85°. Mod. sol. cold EtOH.Aq. Insol.  $H_2O$ .

*Formyl* : leaflets. M.p. 129°. Sol. EtOH,  $C_6H_6$ ,  $CHCl_3$ . Less sol.  $Et_2O$ . Spar. sol. hot  $H_2O$ .

*Acetyl* : 2-acetnaphthalide. Leaflets from  $H_2O$  or EtOH. M.p. 134°. *B, HCl* : needles. M.p. 152°.

*Diacetyl* : plates from ligroin. M.p. 66.5°. Sol. most org. solvents.

*Chloroacetyl* : needles from EtOH.Aq. M.p. 117–18°. Sol. EtOH. Spar. sol.  $C_6H_6$ . Insol.  $H_2O$ .

*Butyryl* : cryst. from pet. ether. M.p. 125°.

*Valeryl* : m.p. 112°.

*Caproyl* : m.p. 107°.

*Heptyl* : m.p. 101°.

*Capryl* : m.p. 103°.

*Pelargonyl* : m.p. 103°.

*Capryl* : m.p. 104°.

*Lauryl* : m.p. 106°.

*Myristyl* : needles from EtOH. M.p. 108°. B.p. 179°/10 mm. Very sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Sol.  $Me_2CO$ .

*Palmityl* : cryst. from EtOH. M.p. 109°. B.p. 198.5°/10 mm. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .

# 1-Naphthylamine-2 : 4-disulphonic Acid 33 1-Naphthylamine-3 : 7-disulphonic Acid

*Stearyl*: needles from EtOH. M.p. 112° (109°). B.p. 220.5°/10 mm. Very sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.

*Benzoyl*: 2-benzonaphthalide. Needles from C<sub>6</sub>H<sub>6</sub> or AcOH. M.p. 162-3°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, hot EtOH. *Benzenesulphonyl*: needles. M.p. 161-2°.

*Benzenesulphonyl*: needles or prisms from EtOH. M.p. 102-3°.

*p-Toluenesulphonyl*: needles and leaflets from EtOH. M.p. 133°.

Knoll, D.R.P., 241,853, (*Chem. Zentr.*, 1912, I, 178).

Knoevenagel, *J. prakt. Chem.*, 1914, 89, 17, *et seq.*

De'Conno, *Gazz. chim. ital.*, 1917, 47, i, 98.

Robertson, *J. Chem. Soc.*, 1919, 115, 1221.

Le Sueur, *J. Chem. Soc.*, 1911, 99, 830.

Pawlewski, *Ber.*, 1902, 35, 112.

Cosiner, *Ber.*, 1881, 14, 59.

Badische, D.R.P., 117,471, (*Chem. Zentr.*, 1901, I, 349).

Campbell, *Chem. Abstracts*, 1922, 16, 3400.

A.G.F.A., D.R.P., 164,130, (*Chem. Zentr.*, 1905, II, 1476).

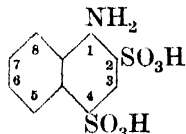
Reilly, Drumm, O'Sullivan, *J. Soc. Chem. Ind.*, 1927, 46, 437T.

Kym, *J. prakt. Chem.*, 1895, 51, 328, 332.

Warren, Smiles, *J. Chem. Soc.*, 1932, 2778.

Ryan, Drumm, *Chem. Abstracts*, 1919, 13, 563.

## 1-Naphthylamine-2 : 4-disulphonic Acid



C<sub>10</sub>H<sub>8</sub>O<sub>6</sub>NS<sub>2</sub>

MW, 303

NaOH fusion → 1-amino-2-naphthol-4-sulphonic acid. Diazo-comp. warmed + dil. HNO<sub>3</sub> → 2 : 4-dinitronaphthol. Di-Na salt : easily sol. H<sub>2</sub>O.

Gulinov, *Chem. Abstracts*, 1933, 27, 3929.

Hunter, Sprung, *J. Am. Chem. Soc.*, 1931, 53, 1437.

## 1-Naphthylamine-2 : 5-disulphonic Acid.

Needles. Very sol. H<sub>2</sub>O. Spar. sol. dil. min. acids. Alkali salts very spar. sol. H<sub>2</sub>O. Sols. of acid and salts fluor. bluish-green. Part. hyd. by dil. H<sub>2</sub>SO<sub>4</sub> at 160° → 1-naphthylamine-5-sulphonic acid. 50% NaOH.Aq. at 240-70° → 5-amino-1-naphthol-6-sulphonic acid.

Sulphonation by 40% anhydro-acid at 120° → 1-naphthylamine-2 : 5 : 7-trisulphonic acid.

Badische, D.R.P., 160,536, (*Chem. Zentr.*, 1905, I, 1678).

Tobias, *Ber.*, 1890, 23, 1631.

## 1-Naphthylamine-2 : 7-disulphonic Acid (*Kalle's Acid*).

Needles. Sols. of alkali salts show weak blue fluor. Heat with dil. H<sub>2</sub>SO<sub>4</sub> or H<sub>2</sub>O under press. → 1-naphthylamine-7-sulphonic acid. Na salt : prisms from dil. EtOH. Ba salt : very spar. sol. H<sub>2</sub>O.

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

## 1-Naphthylamine-2 : 8-disulphonic Acid.

Felted needles. Mod. sol. H<sub>2</sub>O. Sols. of alkali salts show green fluor. Does not give reactive diazo-comp., but couples with diazotised bases → azo-dyestuffs. Fused with NaOH → 8-amino-1-naphthol-7-sulphonic acid.

Dressel, Kothe, *Ber.*, 1894, 27, 2139.

## 1-Naphthylamine-3 : 5-disulphonic Acid.

*Mono-K salt* : needles + 2H<sub>2</sub>O.

Armstrong, Wynne, *Proc. Chem. Soc.*, 1895, 11, 240.

## 1-Naphthylamine-3 : 6-disulphonic Acid (*Freund's Acid*).

Needles. Very sol. H<sub>2</sub>O, EtOH. H<sub>2</sub>O at 180° → 1-naphthol-3 : 6-disulphonic acid. Heat with 75% NaOH.Aq. → 4-amino-2-naphthol-7-sulphonic acid + 5-amino-2-naphthol-7-sulphonic acid. NH<sub>3</sub> at 180° → 1 : 3-naphthylenediamine-6-sulphonic acid. NaHg → 1-naphthylamine. Mono-Na and K salts : cryst. + 3H<sub>2</sub>O. Very sol. H<sub>2</sub>O. Ca salt : cryst. + 5H<sub>2</sub>O. Sol. H<sub>2</sub>O. Ba salt : cryst. + 4H<sub>2</sub>O. Sol. H<sub>2</sub>O.

Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

Friedländer, Taussig, *Ber.*, 1897, 30, 1462.

Armstrong, Wynne, *Proc. Chem. Soc.*, 1895, 11, 82.

Alén, *Ber.*, 1884, 17 (Referate), 436.

## 1-Naphthylamine-3 : 7-disulphonic Acid.

Needles. Very sol. H<sub>2</sub>O. Sols. of the acid and its salts show blue fluor. H<sub>2</sub>O at 180° → 1-naphthol-3 : 7-disulphonic acid. 40% NaOH.Aq. at 200° → 8-amino-2-naphthol-6-sulphonic acid + an isomeric acid. NH<sub>3</sub> at 180° → 1 : 3-naphthylenediamine-7-sulphonic acid. Mono-K salt : needles. Mod. sol. hot H<sub>2</sub>O. Ca salt : needles + 2H<sub>2</sub>O. Insol. cold,

## 1-Naphthylamine-3 : 8-disulphonic Acid 34 2-Naphthylamine-1 : 5-disulphonic Acid

sol. hot  $\text{H}_2\text{O}$ . Ba salt : needles +  $1\text{H}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

See first and fourth references above.

### 1-Naphthylamine-3 : 8-disulphonic Acid.

Flakes +  $3\text{H}_2\text{O}$ . Very sol. hot  $\text{H}_2\text{O}$ . The diazo-comp. + dil.  $\text{H}_2\text{SO}_4$  on warming  $\rightarrow$  naphthasultone-3-sulphonic acid. Couples with diazotised *m*- or *p*-nitroaniline but not with diazotised aniline.  $\text{H}_2\text{O}$  at  $180^\circ$  or prolonged boiling + dil.  $\text{H}_2\text{SO}_4 \rightarrow$  1-naphthol-3 : 8-disulphonic acid. Boiled with 75%  $\text{H}_2\text{SO}_4$ , or with Zn dust +  $\text{NaOH}$ .Aq., or with  $\text{NaHg}$  in cold  $\rightarrow$  1-naphthylamine-3-sulphonic acid. Fused with  $\text{NaOH}$  below  $210^\circ \rightarrow$  1-amino-8-naphthol-sulphonic acid. Heat with 9%  $\text{NaOH}$ .Aq. at  $250^\circ \rightarrow$  1 : 8-dihydroxynaphthalene-3-sulphonic acid.  $\text{NH}_3$  at  $180^\circ \rightarrow$  1 : 3-naphthylene-diamine-8-sulphonic acid. Mono-Na salt : cryst. +  $2\text{H}_2\text{O}$ . Sol. 30 parts  $\text{H}_2\text{O}$ . Di-Na salt : cryst. +  $6\text{H}_2\text{O}$ . Very sol.  $\text{H}_2\text{O}$ . Ba salt : needles +  $4\text{H}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

Stanier, E.P., 161,859, (*Chem. Abstracts*, 1921, 15, 2445).

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

Paul, Z. angew. Chem., 1896, 9, 562.

Friedländer, Lucht, Ber., 1893, 26, 3032.

Schultz, Ber., 1890, 23, 77.

### 1-Naphthylamine-4 : 6-disulphonic Acid (*Dahl's Acid II*).

Needles. Sol. to 17% in  $\text{H}_2\text{O}$  at  $20^\circ$ , very sol. hot  $\text{H}_2\text{O}$ . Sol. hot 85% EtOH. Sols. of acid and salts fluor. blue. Couples with diazo-comps. Fused with  $\text{NaOH}$  at  $180$ – $200^\circ \rightarrow$  1-amino-6-naphthol-4-sulphonic acid; at  $200$ – $20^\circ \rightarrow$  1 : 6-dihydroxynaphthalene-4-sulphonic acid. Sulphonated with 35% anhydro-acid at  $80$ – $90^\circ \rightarrow$  1-naphthylamine-2 : 4 : 6-trisulphonic acid. Mono-Na salt : sol. 6 parts  $\text{H}_2\text{O}$  at  $20^\circ$ . Di-Na and K salts : very sol.  $\text{H}_2\text{O}$ . Ca salt : needles +  $5\text{H}_2\text{O}$ .

Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

Erdmann, Ann., 1893, 275, 218.

Armstrong, Wynne, Proc. Chem. Soc., 1890, 6, 126.

### 1-Naphthylamine-4 : 7-disulphonic Acid (*Dahl's Acid III*).

Needles from  $\text{H}_2\text{O}$ . Sol. to 0.7%  $\text{H}_2\text{O}$  at  $20^\circ$ , to 5% at  $100^\circ$ . Insol. 85% EtOH. Sols. of acid and its salts show blue fluor. Couples with diazo-comps. Sulphonated with 35% anhydro-acid at  $80$ – $90^\circ \rightarrow$  1-naphthylamine-2 : 4 : 7-

trisulphonic acid. Mono-Na salt : needles, Sol. 140 parts  $\text{H}_2\text{O}$  at  $20^\circ$ , 20 parts at b.p. Insol. 85% EtOH. Di-K salt : cryst. +  $2\frac{1}{2}\text{H}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ . Ca salt : spar. sol. cold  $\text{H}_2\text{O}$ .

Bucherer, Barsch, J. prakt. Chem., 1925, 111, 322.

M.L.B., D.R.P., 215,338, (*Chem. Zentr.*, 1909, II, 1710).

See also second reference above.

### 1-Naphthylamine-4 : 8-disulphonic Acid.

Couples with diazo-comps. Fused with  $\text{NaOH}$  at  $200^\circ \rightarrow$  1-amino-8-naphthol-4-sulphonic acid. 60%  $\text{NaOH}$ .Aq. at  $250^\circ \rightarrow$  1 : 8-dihydroxynaphthalene-4-sulphonic acid. Sulphonated with 40% anhydro-acid  $\rightarrow$  naphthasultam-2 : 4-disulphonic acid. Mono-Na salt : flakes. Spar. sol. cold  $\text{H}_2\text{O}$ . Di-Na salt : needles +  $2\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ .

König, Keil, Ber., 1922, 55, 2149.

Stanier, E.P., 161,859, (*Chem. Abstracts*, 1921, 15, 2445).

Kalle, D.R.P., 283,727, (*Chem. Zentr.*, 1915, I, 1238).

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

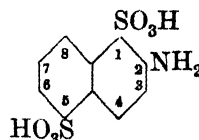
Bucherer, J. prakt. Chem., 1907, 75, 258; 1904, 70, 347.

### 1-Naphthylamine-5 : 8-disulphonic Acid.

Needles. Very spar. sol.  $\text{H}_2\text{O}$ . Sols. of alkali salts show intense greenish-yellow col. Couples with diazo-comps. 75%  $\text{NaOH}$ .Aq. at  $150$ – $60^\circ \rightarrow$  1-amino-8-naphthol-5-sulphonic acid; 60%  $\text{NaOH}$ .Aq. at  $250^\circ \rightarrow$  1 : 8-dihydroxynaphthalene-4-sulphonic acid.  $\text{NaHg} \rightarrow$  1-naphthylamine. Sulphonation with 40% anhydro-acid at  $80$ – $90^\circ \rightarrow$  naphthasultam-disulphonic acid-D. Mono-Na salt : needles +  $1\frac{1}{2}\text{H}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

Gattermann, Ber., 1899, 32, 1156.

### 2-Naphthylamine-1 : 5-disulphonic Acid



$\text{C}_{10}\text{H}_6\text{O}_6\text{NS}_2$

MW, 303

The diazo-comp. + alk.  $\text{NaOCl}$  or +  $\text{Na}_2\text{CO}_3$  at  $50$ – $60^\circ \rightarrow$  diazo-oxide of 2-amino-1-naphthol-5-sulphonic acid. Fused with  $\text{NaOH}$  at  $210$ – $30^\circ \rightarrow$  2-amino-5-naphthol-1-sulphonic acid. Sulphonation with 40% anhydro-acid at  $100^\circ \rightarrow$  2-naphthylamine 1 : 5 : 7-trisulphonic acid.

## 2-Naphthylamine-1 : 6-disulphonic Acid 35 2-Naphthylamine-5 : 7-disulphonic Acid

*Di-K salt* : cryst. Mod. sol.  $\text{H}_2\text{O}$ .

Bucherer, Wahl, *J. prakt. Chem.*, 1921, 103, 129.

Kalle, D.R.P., 233,105, (*Chem. Zentr.*, 1911, I, 1263).

Armstrong, Wynne, *Proc. Chem. Soc.*, 1896, 12, 238; 1890, 6, 129.

### 2-Naphthylamine-1 : 6-disulphonic Acid.

Needles. Very sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Dil. sols. show weak blue fluor. The cryst. diazo-comp. +  $\text{Na}_2\text{CO}_3$ .Aq.  $\rightarrow$  diazo-oxide of 2-amino-1-naphthol-6-sulphonic acid. Mono-Na salt : long needles +  $2\text{H}_2\text{O}$ . Very sol.  $\text{H}_2\text{O}$ . Mono-K salt : needles +  $1\text{H}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ . Di- $\text{NH}_4$  salt : red prisms +  $1\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ . Di-Na salt : long needles. Very sol.  $\text{H}_2\text{O}$ . Di-K salt : large yellow cryst. +  $2\text{H}_2\text{O}$ . Very sol.  $\text{H}_2\text{O}$ .

Badische, D.R.P., 148,882, (*Chem. Zentr.*, 1904, I, 619).

Armstrong, Wynne, *Proc. Chem. Soc.*, 1890, 6, 130.

Forsling, *Ber.*, 1888, 21, 3495.

### 2-Naphthylamine-1 : 7-disulphonic Acid.

Sols. of salts show violet-blue fluor. In AcOH does not couple with diazobenzene. Long boiling with 20% HCl  $\rightarrow$  2-naphthylamine-7-sulphonic acid. The diazo-comp. +  $\text{Na}_2\text{CO}_3$ .Aq.  $\rightarrow$  diazo-oxide of 2-amino-1-naphthol-7-sulphonic acid. Mono-K salt : needles. Spar. sol. cold  $\text{H}_2\text{O}$ . Di-K salt : large cryst. +  $3\text{H}_2\text{O}$ . Ba salt : needles. Mod. sol.  $\text{H}_2\text{O}$ .

Dressel, Kothe, *Ber.*, 1894, 27, 1194.

Armstrong, Wynne, *Chem. News*, 1890, 62, 164.

### 2-Naphthylamine-3 : 6-disulphonic Acid (*Amido-R-acid*).

Plates or amorph. powder. Sol.  $\text{H}_2\text{O}$  with violet-blue fluor. Couples with diazo-comps. 75% NaOH.Aq. at  $230-50^\circ \rightarrow$  2-amino-3-naphthol-6-sulphonic acid.

Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

Griesheim-Elektron Fabrik, D.R.P., 217,277, (*Chem. Zentr.*, 1910, I, 395).

Armstrong, Wynne, *Proc. Chem. Soc.*, 1890, 6, 12.

Pfützing, Duisberg, *Ber.*, 1889, 22, 398.

### 2-Naphthylamine-3 : 7-disulphonic Acid.

Cryst. Sol. hot, spar. sol. cold  $\text{H}_2\text{O}$ . Couples with diazo-comps. Dil. sols. of alkali salts show blue fluor. Sulphonation with 40% anhydro-acid at  $80-90^\circ \rightarrow$  2-naphthylamine-1 : 3 : 7-trisulphonic acid. Mono-Na salt : sol. 50 parts  $\text{H}_2\text{O}$

at  $20^\circ$ , 12.5 parts at b.p. Mono-K salt : flakes. Spar. sol.  $\text{H}_2\text{O}$ . Acid Ba salt : microscopic flakes. Very spar. sol.  $\text{H}_2\text{O}$ .

Dressel, Kothe, *Ber.*, 1894, 27, 1199.

Armstrong, Wynne, *Proc. Chem. Soc.*, 1890, 6, 127.

### 2-Naphthylamine-4 : 7-disulphonic Acid (*Andresen's Acid*).

Dil. sols. of alkali salts fluor. strongly blue. The diazo-comp. boiled with  $\text{H}_2\text{O} \rightarrow$  2-naphthol-4 : 7-disulphonic acid. 35% KOH.Aq. at  $180-200^\circ \rightarrow$  2-amino-4-naphthol-7-sulphonic acid. Mono-Na salt : needles +  $1\text{H}_2\text{O}$  from boiling  $\text{H}_2\text{O}$ . Ba salt : prisms from boiling  $\text{H}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

Armstrong, Wynne, *Proc. Chem. Soc.*, 1891, 7, 27.

Schultz, *Ber.*, 1890, 23, 77.

See also first reference above.

### 2-Naphthylamine-4 : 8-disulphonic Acid.

Prisms. The alkali salts in aq. sol. show deep blue fluor. Does not couple with diazo-comps.  $\text{H}_2\text{O}$  or 10%  $\text{H}_2\text{SO}_4$  at  $180^\circ \rightarrow$  2-naphthol-4-sulphonic acid. Fused with KOH at  $215^\circ \rightarrow$  2-amino-4-naphthol-8-sulphonic acid. The acetyl deriv. +  $\text{HNO}_3 \rightarrow$  6-nitro-deriv. Boiled with Zn dust + NaOH.Aq.  $\rightarrow$  2-naphthylamine-4-sulphonic acid + 2-naphthylamine-8-sulphonic acid. NaHg in cold  $\rightarrow$  2-naphthylamine-8-sulphonic acid + 2-naphthylamine. Na salt : needles. Sol. hot  $\text{H}_2\text{O}$ . Acid Ba salt : minute needles. Sol.  $\text{H}_2\text{O}$ .

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

Friedländer, Fischer, *Chem. Zentr.*, 1899, I, 289.

Friedländer, Lucht, *Ber.*, 1893, 26, 3033.

### 2-Naphthylamine-5 : 7-disulphonic Acid.

Rhombic needles +  $5\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . Sol. to 23% in  $\text{H}_2\text{O}$  at  $20^\circ$ . Sols. of salts show green fluor. 50% NaOH.Aq. at  $190^\circ \rightarrow$  2-amino-5-naphthol-7-sulphonic acid. NaHg in cold  $\rightarrow$  2-naphthylamine-7-sulphonic acid. Sulphonation with 40% anhydro-acid at  $100^\circ \rightarrow$  2-naphthylamine-1 : 5 : 7-trisulphonic acid. Mono-Na salt : needles +  $4\text{H}_2\text{O}$ . Sol. to 8% in  $\text{H}_2\text{O}$  at  $20^\circ$ . Mono-K salt : prisms +  $4\text{H}_2\text{O}$ . Sol. to 2.6% in  $\text{H}_2\text{O}$  at  $20^\circ$ . Di-Na salt : needles +  $6\text{H}_2\text{O}$ . Sol. to 72% in  $\text{H}_2\text{O}$  at  $20^\circ$ . Di-K salt : needles +  $2\text{H}_2\text{O}$ . Sol. to 64% in  $\text{H}_2\text{O}$  at  $20^\circ$ . Ca salt : hexagonal cryst. +  $4\text{H}_2\text{O}$ . Sol. to 40% in  $\text{H}_2\text{O}$  at  $20^\circ$ . Ba salt : cryst. +  $3\text{H}_2\text{O}$ .

Sol. to 23% in  $H_2O$  at  $20^\circ$ . Mg salt : needles or prisms +  $8H_2O$ . Sol. to 21% in  $H_2O$  at  $20^\circ$ .

Ofitzerov, *Chem. Abstracts*, 1935, **29**, 2530, 1086.

Fierz-David, Braunschweig, *Helv. Chim. Acta*, 1923, **6**, 1146.

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

Dressel, Kothe, *Ber.*, 1894, **27**, 1197.

Armstrong, Wynne, *Proc. Chem. Soc.*, 1890, **6**, 129.

### 2-Naphthylamine-6 : 8-disulphonic Acid (Amido-G-Acid).

Needles +  $4H_2O$  from  $H_2O$ . Sol. to 9% in  $H_2O$  at  $20^\circ$ . Sols. of salts show blue fluor. Does not couple with diazo-comps. 75% NaOH.Aq. at  $230-50^\circ \rightarrow$  2-amino-8-naphthol-6-sulphonic acid ("Gamma-acid"). Sulphonation with 40% anhydro-acid at  $120-30^\circ \rightarrow$  2-naphthylamine-3 : 6 : 8-trisulphonic acid. Mono-K salt : rhombic cryst. +  $2H_2O$ . Sol. to 2.5% in  $H_2O$  at  $20^\circ$ . Di-Na salt : prisms +  $3H_2O$ . Sol. to 59% in  $H_2O$  at  $20^\circ$ . Di-K salt : prisms +  $2H_2O$ . Sol. to 52% in  $H_2O$  at  $20^\circ$ . Ca salt : plates +  $3H_2O$ . Sol. to 29% in  $H_2O$  at  $20^\circ$ . Ba salt : plates +  $3H_2O$ . Sol. to 12% in  $H_2O$  at  $20^\circ$ . Mg salt : cryst. +  $8H_2O$ . Sol. to 9% in  $H_2O$  at  $20^\circ$ .

Veinberg, *Chem. Abstracts*, 1935, **29**, 8336.

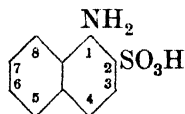
Bucherer, *J. prakt. Chem.*, 1904, **70**, 358.

Dressel, Kothe, *Ber.*, 1894, **27**, 2152.

Armstrong, Wynne, *Proc. Chem. Soc.*, 1890, **6**, 128.

See also first two references above.

### 1-Naphthylamine-2-sulphonic Acid



$C_{10}H_9O_3NS$

MW, 223

Needles from  $H_2O$ . M.p.  $272^\circ$  decomp. Sol. 244 parts  $H_2O$  at  $20^\circ$ ; 31.3 parts at  $100^\circ$ . Heat of comb.  $\bar{C}_p$  1258.1 Cal.  $HNO_3 \rightarrow$  5-nitro-deriv. Sulphonation with 10% anhydro-acid in cold  $\rightarrow$  1-naphthylamine 2 : 5-disulphonic acid. Na salt : flakes. Sol. 10 parts boiling  $H_2O$ , 60 parts cold  $H_2O$ . K salt : needles. Spar. sol. cold  $H_2O$ . Ca salt : flakes. Spar. sol.  $H_2O$ .

N-Acetyl : needles +  $1H_2O$  from  $H_2O$ .

N-Et : needles from  $H_2O$ . M.p.  $207-8^\circ$ . Spar. sol.  $H_2O$ . Insol.  $C_6H_6$ .

Bayer, D.R.P., 293,184, (*Chem. Zentr.*, 1916, II, 291).

Wildt, *Rec. trav. chim.*, 1904, **23**, 185.

Erdmann, *Ann.*, 1893, **275**, 225.

### 1 - Naphthylamine - 3 - sulphonic Acid (Cleve's $\gamma$ -Acid).

Needles. Spar. sol.  $H_2O$ . Couples with diazo-comps. Fused with KOH at  $250-60^\circ \rightarrow$  1-amino-3-naphthol. 60% NaOH.Aq. at  $250-80^\circ \rightarrow$  o-toluic acid.  $NH_3$ .Aq. at  $180^\circ \rightarrow$  1 : 3-naphthylenediamine. Sulphonation with 20% anhydro-acid in cold  $\rightarrow$  1-naphthylamine-3 : 5-disulphonic acid. Na, K, Ca and Ba salts : very sol.  $H_2O$ .

N-Phenyl :  $C_{16}H_{13}O_3NS$ . MW, 299. Cryst. Mod. sol.  $H_2O$ . Na salt : mod. sol.  $H_2O$ .

Amide :  $C_{10}H_{10}O_2N_2S$ . MW, 222. Needles +  $1H_2O$ . M.p.  $131^\circ$ . 1-N-Acetyl : m.p.  $220-1^\circ$ .

Bayer, D.R.P., 255,724; 251,099, (*Chem. Zentr.*, 1913, I, 478; 1912, II, 1243); 70, 349.

Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

Gattermann, Schulze, *Ber.*, 1897, **30**, 54.

Friedländer, Lucht, *Ber.*, 1893, **26**, 3032.

### 1-Naphthylamine-4-sulphonic Acid.

See Naphthionic Acid.

### 1 - Naphthylamine - 5 - sulphonic Acid (Laurent's Acid).

Needles +  $1H_2O$ . Sol. 940 parts cold  $H_2O$ . Heat of comb.  $\bar{C}_p$  1255.2 Cal.  $k = 2.4 \times 10^{-4}$  at  $25^\circ$ . Dil. aq. sols. of the acid and its salts show greenish fluor. Diazo-comp. forms first component of many azo-dyestuffs. Conc. NaOH.Aq. at  $250^\circ \rightarrow$  1-amino-5-naphthol. Sulphonation with 35% anhydro-acid at  $90-120^\circ \rightarrow$  1-naphthylamine-2 : 5 : 7-trisulphonic acid. The acetyl deriv. + 30% anhydro-acid in cold  $\rightarrow$  the 5 : 7-disulphonic acid. Na salt : needles. Sol.  $H_2O$ . K salt : needles +  $1H_2O$ . Sol.  $H_2O$ . Ca salt : plates +  $9H_2O$ . Sol.  $H_2O$ .

N-Phenyl : cryst. Spar. sol.  $H_2O$ . Na salt : spar. sol.  $H_2O$ .

N-Acetyl : aniline salt, decomp. at  $344^\circ$ .

Amide : m.p.  $259-60^\circ$ . 1-N-Acetyl : m.p.  $231-2^\circ$ . 1 : 1-N-Diacetyl : m.p.  $200^\circ$ .

Anilide : m.p.  $171^\circ$ .

Vendelshtein, Shpinel, *Chem. Abstracts*, 1933, **27**, 5320.

Newport Chem. Co., D.R.P., 562,513, (*Chem. Abstracts*, 1933, **27**, 1010).

Heller, Sturm, *J. prakt. Chem.*, 1929, **121**, 193.

Forster et al., *J. Soc. Chem. Ind.*, 1928, **47**, 155, 157r.

Fierz, *Helv. Chim. Acta*, 1920, **3**, 318.

Erdmann, *Ann.*, 1893, **275**, 193, 264.

Bayer, D.R.P., 70,349.

**1 - Naphthylamine - 6 - sulphonic Acid**  
(*Cleve's Acid*).

Needles + 2H<sub>2</sub>O. Sol. 1000 parts H<sub>2</sub>O at 16°. Heat of comb.  $C_p$  1258.4 Cal.  $k = 1.95 \times 10^{-4}$  at 25°. Couples with diazo-comps. Fused with KOH  $\rightarrow$  1-amino-6-naphthol. Sulphonation with 10% anhydro-acid  $\rightarrow$  1-naphthylamine-4 : 6-disulphonic acid. Na salt : plates +  $4\frac{1}{2}$  H<sub>2</sub>O. Sol. H<sub>2</sub>O. K salt : flakes + 1H<sub>2</sub>O. Sol. H<sub>2</sub>O. Ba salt : needles + 1H<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Ca salt : plates + 7H<sub>2</sub>O. Sol. H<sub>2</sub>O.

N-Phenyl : cryst. Spar. sol. H<sub>2</sub>O. Na salt : spar. sol. H<sub>2</sub>O.

Amide : m.p. 218-19° decomp. 1-N-Acetyl : m.p. 238-9°.

Anilide : m.p. 127-8°.

See last five references above.

**1 - Naphthylamine - 7 - sulphonic Acid**  
(*Cleve's-θ-Acid*).

Flakes + 1H<sub>2</sub>O. Sol. 220 parts H<sub>2</sub>O at 25°. Couples with diazo-comps. Heat of comb.  $C_p$  1259.5 Cal.  $k = 2.27 \times 10^{-4}$  at 25°. FeCl<sub>3</sub>  $\rightarrow$  blue col. 60% NaOH.Aq. at 250°  $\rightarrow$  1-amino-7-naphthol. Sulphonated with 25% anhydro-acid at 50°  $\rightarrow$  1-naphthylamine-4 : 7-disulphonic acid. Na salt : needles +  $\frac{1}{2}$  H<sub>2</sub>O. Sol. H<sub>2</sub>O. K salt : flakes. Sol. H<sub>2</sub>O. Ba salt : needles. Spar. sol. H<sub>2</sub>O. Ca salt : cryst. + 2H<sub>2</sub>O. Sol. H<sub>2</sub>O.

N-Phenyl : cryst. Spar. sol. H<sub>2</sub>O. Na salt : mod. sol. H<sub>2</sub>O.

Amide : needles +  $1\frac{1}{2}$  H<sub>2</sub>O. M.p. 181°. 1-N-Acetyl : m.p. 213°.

Anilide : m.p. 146-7°.

Friedländer, Zinberg, *Ber.*, 1896, **29**, 41.

Cleve, *Ber.*, 1888, **21**, 3264.

Bayer, D.R.P., 70,349.

A.G.F.A., D.R.P., 159,353, (*Chem. Zentr.*, 1905, I, 975).

See also previous references.

**1-Naphthylamine-8-sulphonic Acid** (*Peri Acid*, *Schöllkopf Acid*).

Needles + 1H<sub>2</sub>O. Sol. 4800 parts H<sub>2</sub>O at 21°, 238 parts boiling H<sub>2</sub>O. Heat of comb.  $C_p$  1254.4 Cal.  $k = 1.02 \times 10^{-5}$  at 25°. Couples with diazo-comps. H<sub>2</sub>O at 200°  $\rightarrow$  1-naphthol-8-sulphonic acid. 9% NaOH.Aq. at 220-60°  $\rightarrow$  1 : 8-dihydroxynaphthalene. Fused with KOH at 200°  $\rightarrow$  1-amino-8-naphthol. POCl<sub>3</sub> at 130°  $\rightarrow$  naphthasultam. 40% anhydro-acid at 80-90°  $\rightarrow$  naphthasultam-2 : 4-disulphonic acid; 10% anhydro-acid in cold  $\rightarrow$  1-naphthylamine-4 : 8-disulphonic acid.

N-Acetyl : aniline salt, m.p. 273°.

Anilide : m.p. 139-40°.

Chuksanova, Bilik, *Chem. Abstracts*, 1935, **29**, 1085.

Mow, U.S.P., 1,996, 822, (*Chem. Abstracts*, 1935, **29**, 3353).

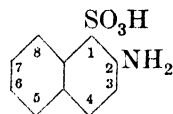
Tinker, E.P., 389,098, (*Chem. Abstracts*, 1933, **27**, 4545).

Martin, *Chem. Abstracts*, 1928, **22**, 2665.

Forster, Hanson, Watson, *J. Soc. Chem. Ind.*, 1928, **47**, 155r.

Wahl, Vermeylen, *Bull. soc. chim.*, 1927, **41**, 514.

Finzi, *Ann. chim. applicata*, 1925, **14**, 50.

**2 - Naphthylamine - 1 - sulphonic Acid**  
(*Tobias' Acid*)

C<sub>10</sub>H<sub>9</sub>O<sub>3</sub>NS

MW, 223

Anhyd. flakes from hot, or hydrated needles from cold H<sub>2</sub>O. Used as first component in many azo-dyestuffs. Diazo-comp. readily forms 2-naphthol-1-sulphonic acid. Na salt heated in dry atm. at 230°  $\rightarrow$  Na β-naphthylsulphamate. Sulphonation with 20% anhydro-acid  $\rightarrow$  2-naphthylamine-1 : 5-disulphonic acid.

Green, Vakil, *J. Chem. Soc.*, 1918, **113**, 40.

Bucherer, *J. prakt. Chem.*, 1904, **70**, 357.

Dressel, Kothe, *Ber.*, 1894, **27**, 2140.

Erdmann, *Ann.*, 1893, **275**, 274.

**2-Naphthylamine-4-sulphonic Acid.**

Long needles + 1H<sub>2</sub>O. Couples with diazo-comps. 60% NaOH.Aq. at 230-80°  $\rightarrow$  o-toluic acid. Na salt : flakes + 4H<sub>2</sub>O. Aq. sols. show violet fluor. K salt : needles +  $1\frac{1}{2}$  H<sub>2</sub>O.

Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

Friedländer, Rüdte, *Ber.*, 1896, **29**, 1611.

**2-Naphthylamine-5-sulphonic Acid.**

Needles from H<sub>2</sub>O. Sol. 0.033 in 100 parts H<sub>2</sub>O at 20°, 1 in 260 parts boiling H<sub>2</sub>O.  $k = 9.4 \times 10^{-5}$  at 25°. The acid and its salts in dil. aq. sol. show reddish-blue fluor. KOH at 260-70°  $\rightarrow$  2-amino-5-naphthol. NaHg  $\rightarrow$  2-naphthylamine + SO<sub>2</sub>. Sulphonation with 20% anhydro-acid in cold  $\rightarrow$  2-naphthylamine-5 : 7-disulphonic acid (mainly) + 2-naphthylamine-1 : 5-disulphonic acid. Na salt : prisms + 5H<sub>2</sub>O. Sol. 10 parts boiling 95% EtOH. The aq. sol. + CuSO<sub>4</sub>  $\rightarrow$  deep red col. Ca salt : needles + 11H<sub>2</sub>O. Sol. 11 parts cold H<sub>2</sub>O.

N-Phenyl : C<sub>16</sub>H<sub>13</sub>O<sub>3</sub>NS. MW, 299. Cryst.

## 2-Naphthylamine-6-sulphonic Acid

38

Very spar. sol.  $\text{H}_2\text{O}$ . *Na*, *K*, and  $\text{NH}_4$  salts mod. sol.  $\text{H}_2\text{O}$ .

*Amide*:  $\text{C}_{10}\text{H}_{10}\text{O}_2\text{N}_2\text{S}$ . MW, 222. Needles. M.p. 165°.

Waterman, Groot, *Chem. Zentr.*, 1928, I, 1288.

Green, Vakil, *J. Chem. Soc.*, 1918, 113, 38.

Kappeler, *Ber.*, 1912, 45, 635.

Erdmann, *Ann.*, 1893, 275, 277.

Friedländer, Lucht, *Ber.*, 1893, 26, 3032.

Lesser, *Ber.*, 1894, 27, 2364.

Clayton Aniline, D.R.P., 53,649.

## 2-Naphthylamine-6-sulphonic Acid (Brønner Acid).

Leaflets +  $1\text{H}_2\text{O}$ . Sol. 7790 parts  $\text{H}_2\text{O}$  at 20°, 630 parts boiling  $\text{H}_2\text{O}$ . Sols of acid or its salts show bluish fluor. Diazotises and couples with phenols and amines. Sulphonated with 20% anhydro-acid at 20°  $\rightarrow$  2-naphthylamine-1:6-disulphonic acid + 2-naphthylamine-6:8-disulphonic acid.  $\text{NH}_4$  salt: plates +  $1\text{H}_2\text{O}$ . Very spar. sol.  $\text{H}_2\text{O}$ . *Na* salt: cryst. +  $2\text{H}_2\text{O}$ . Sol. 40 parts cold  $\text{H}_2\text{O}$ . Aq. sol. +  $\text{CuSO}_4 \rightarrow$  yellow ppt.  $\text{K}_3\text{Fe}(\text{CN})_6$  in alk. sol.  $\rightarrow$  dinaphthazine-disulphonic acid. *K* salt: cryst. +  $2\text{H}_2\text{O}$ . Sol. 45 parts cold  $\text{H}_2\text{O}$ . *Ca* salt, *Ba* salt: each +  $6\text{H}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

*N-Phenyl*: plates. Sol. 15 parts hot, 60 parts cold  $\text{H}_2\text{O}$ . *Na* salt: sol. 22 parts hot  $\text{H}_2\text{O}$ .

*N-Acetyl*: aniline salt, m.p. 256°.

Forster, Hanson, Watson, *J. Soc. Chem. Ind.*, 1928, 47, 155r.

Reitzenstein, Fitzgerald, *J. prakt. Chem.*, 1914, 89, 288.

Bucherer, *J. prakt. Chem.*, 1904, 70, 357.

Bischoff, *Ber.*, 1890, 23, 1914.

Green, *J. Chem. Soc.*, 1889, 55, 37; 1918, 113, 35.

Bucherer, Stohmann, *J. prakt. Chem.*, 1905, 71, 435, 449.

Badische, D.R.P., 122,570, (*Chem. Zentr.*, 1901, II, 670).

See also first reference above.

## 2-Naphthylamine-7-sulphonic Acid (Bayer's Acid, Amido-F Acid, $\delta$ -Acid).

Needles +  $1\text{H}_2\text{O}$ . Sol. 5040 parts  $\text{H}_2\text{O}$  at 20°, 350 parts boiling  $\text{H}_2\text{O}$ .  $k = 1.02 \times 10^{-4}$  at 25°. Sols. of acid and its salts show reddish-violet fluor. Couples with diazo-comps. 50%  $\text{NaOH}$ . Aq. at 260–80°  $\rightarrow$  2-amino-7-naphthol. The *Na* salt + 20%  $\text{NaHSO}_3$  at 90–100° for 24 hours  $\rightarrow$  *Na* 2:2'-dinaphthylamine-7:7'-disulphonate. Sulphonation with 20–25% anhydro-acid at ord. temp.  $\rightarrow$  1:7-, 4:7-, and 5:7- $\beta$ -naphthyl-

## 2-Naphthylamine-1:3:6:7-tetra-sulphonic Acid

aminedisulphonic acids. *Na* salt: needles +  $4\text{H}_2\text{O}$ . Sol. 70 parts cold  $\text{H}_2\text{O}$ . Aq. sol. +  $\text{CuSO}_4 \rightarrow$  orange-yellow ppt.

*N-Phenyl*: cryst. Spar. sol.  $\text{H}_2\text{O}$ . *Na* salt: spar. sol.  $\text{H}_2\text{O}$ .

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478); 70, 349.

Erdmann, *Ber.*, 1888, 21, 637.

Weinberg, *Ber.*, 1887, 20, 2908.

Bayer, Duisberg, *ibid.*, 1432.

See also third, fourth, and fifth references above.

## 2-Naphthylamine-8-sulphonic Acid (Badische Acid).

Prisms or needles from  $\text{H}_2\text{O}$ . Sol. 1680 parts  $\text{H}_2\text{O}$  at 20°, 200 parts boiling  $\text{H}_2\text{O}$ .  $k = 1.22 \times 10^{-4}$  at 25°. Dil. aq. sols. of the acid and its salts show blue fluor. Aq. sol. +  $\text{CuSO}_4 \rightarrow$  orange-red ppt. With diazo-comps.  $\rightarrow$  sol. yellow diazoamino-comps. Fused with  $\text{KOH}$  at 260–70°  $\rightarrow$  2-amino-8-naphthol.  $\text{NaHg} \rightarrow$  2-naphthylamine +  $\text{SO}_2$ . Sulphonation with 20% anhydro-acid in cold  $\rightarrow$  2-naphthylamine-6:8-disulphonic acid. *K* salt: six-sided plates +  $\frac{1}{2}\text{H}_2\text{O}$ . *Ca* salt: plates +  $6\text{H}_2\text{O}$ . Sol. 11 parts cold  $\text{H}_2\text{O}$ .

*N-Phenyl*: *Na*,  $\text{NH}_4$ , and *Ca* salts sol.  $\text{H}_2\text{O}$ .

Green, *J. Soc. Chem.*, 1918, 113, 41; 1889, 55, 36.

Kappeler, *Ber.*, 1912, 45, 635.

Bucherer, *J. prakt. Chem.*, 1904, 70, 358.

Erdmann, *Ann.*, 1893, 275, 281.

Friedländer, Lucht, *Ber.*, 1893, 26, 3033.

Pfizinger, Duisberg, *Ber.*, 1889, 22, 397.

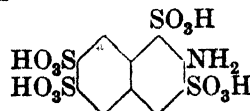
Clayton Aniline, D.R.P., 53, 649.

Bayer, D.R.P., 70,349.

Bucherer, Stohmann, *J. prakt. Chem.*, 1905, 71, 451.

See also first reference above.

## 2-Naphthylamine-1:3:6:7-tetrasul-phonic Acid



$\text{C}_{10}\text{H}_9\text{O}_{12}\text{NS}_4$

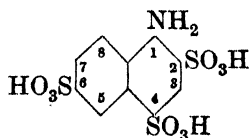
MW, 463

Acid sols. part. hyd. by boiling  $\rightarrow$  2-naphthylamine-3:6:7-trisulphonic acid. Salts in aq. sol. show violet-blue fluor. *Na* salt: cryst. Easily sol.  $\text{H}_2\text{O}$ . *Ba* salt: micro-cryst. +  $6\text{H}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

Dressel, Kothe, *Ber.*, 1894, 27, 1203.

**1-Naphthylamine-2 : 4 : 6-trisulphonic Acid**

39

**1-Naphthylamine-2 : 4 : 6-trisulphonic Acid** $C_{10}H_9O_3NS_3$ 

MW, 383

Does not couple with diazo-comps. Heat with min. acids  $\rightarrow$  1-naphthylamine-2:6-disulphonic acid. NaHg in cold  $\rightarrow$  1-naphthylamine-2 : 4-disulphonic acid. Mono-Na salt : needles. Aq. sols. show blue fluor.

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

**1-Naphthylamine-2 : 4 : 7-trisulphonic Acid.**

Sol. 18.4 in 100 parts  $H_2O$  at  $20^\circ$ , 31.5 in 100 parts  $H_2O$  at  $80^\circ$ . Salts +  $H_2O$  under press. at  $230^\circ \rightarrow$  1-naphthylamine-2 : 7-disulphonic acid. Di-Na salt : cryst. +  $1\frac{1}{2}H_2O$ . Sol. 18.5 in 100 parts  $H_2O$  at  $20^\circ$ , 27.1 in 100 parts at  $80^\circ$ . Di-K salt : cryst. +  $1\frac{1}{2}H_2O$ . Sol. 29.3 in 100 parts  $H_2O$  at  $20^\circ$ , 66.3 in 100 parts at  $80^\circ$ . Ba salt : cryst. +  $3H_2O$ . Sol. 2.5 in 100 parts  $H_2O$  at  $20^\circ$ , 25.7 in 100 parts at  $80^\circ$ . Ca salt : cryst. +  $2H_2O$ . Sol. 16.9 in 100 parts  $H_2O$  at  $20^\circ$ , 32.0 in 100 parts at  $80^\circ$ . Mg salt : cryst. +  $1\frac{1}{2}H_2O$ . Sol. 19.3 in 100 parts  $H_2O$  at  $20^\circ$ , 23.5 in 100 parts at  $80^\circ$ .

Frish, *Helv. Chim. Acta*, 1930, 13, 768.

M.L.B., D.R.P., 215,338, (*Chem. Zentr.*, 1909, II, 1710).

**1-Naphthylamine-2 : 5 : 7-trisulphonic Acid.**

The acid and its Na salt in aq. sol. show green fluor. 50% KOH.Aq. at  $180-200^\circ \rightarrow$  1-amino-5-naphthol-2 : 7-disulphonic acid. NaHg (cold) or Zn dust + boiling NaOH.Aq.  $\rightarrow$  1-naphthylamine-2 : 7-disulphonic acid. Mono-Na salt : needles. Easily sol.  $H_2O$ .

Bayer, D.R.P., 255,724, (*Chem. Zentr.*, 1913, I, 478).

Cassella, D.R.P., 188,505, (*Chem. Zentr.*, 1907, II, 1467).

**1-Naphthylamine-3 : 5 : 7-trisulphonic Acid.**

Salts sol.  $H_2O$  with green fluor. Fused with NaOH at  $160-70^\circ \rightarrow$  1-amino-5-naphthol-3 : 7-disulphonic acid. NaHg (cold) or Zn dust + boiling NaOH.Aq.  $\rightarrow$  1-naphthylamine-3 : 7-disulphonic acid.

See first reference above.

**2-Naphthylamine-1 : 3 : 7-trisulphonic Acid****1-Naphthylamine-3 : 6 : 8-trisulphonic Acid (Koch Acid).**

Hair-like needles +  $6H_2O$  from  $H_2O$ . Sol. 1 in 0.5 parts  $H_2O$  at  $18^\circ$ , 1 in 8 parts EtOH at  $18^\circ$ .  $H_2O$  at  $180-250^\circ \rightarrow$  1-naphthol-3 : 6 : 8-trisulphonic acid. 30-40% NaOH.Aq. at  $210^\circ \rightarrow$  1-amino-8-naphthol-3 : 6-disulphonic acid.  $NH_3$  at  $160-80^\circ \rightarrow$  1 : 3-naphthylenediamine-6 : 8-disulphonic acid. Zn dust + boiling NaOH.Aq.  $\rightarrow$  1-naphthylamine-3 : 6-disulphonic acid. Sulphonation with 25% anhydro-acid at  $70-80^\circ \rightarrow$  naphthasultam-3 : 4 : 6-trisulphonic acid. Di-Na salt : cryst. +  $4H_2O$ . Sol. 1 in 15 parts  $H_2O$  at  $18^\circ$ . Spar. sol. brine, dil. HCl. Tri-Na salt : cryst. +  $3H_2O$ . Sol. 1 in 3 parts  $H_2O$  at  $18^\circ$ . Tri-K salt : cryst. +  $1H_2O$ . Sol. 1 in 4 parts  $H_2O$  at  $18^\circ$ . Ca salt : sol. 1 in 3 parts  $H_2O$  at  $15^\circ$ . Aniline salt : m.p.  $312^\circ$  decomp.

Kurochkin, *Chem. Abstracts*, 1932, 26, 4326.

Forster, Hanson, Watson, *J. Soc. Chem. Ind.*, 1928, 47, 157.

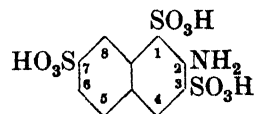
Kalle, D.R.P., 233,934, (*Chem. Zentr.*, 1911, I, 1468).

Dressel, Kothe, *Ber.*, 1894, 27, 2147.

**1-Naphthylamine-4 : 6 : 8-trisulphonic Acid.**

Does not couple with diazo-comps. Boiled with 75%  $H_2SO_4 \rightarrow$  1-naphthylamine-6 : 8-disulphonic acid.  $H_2O$  at  $160-220^\circ \rightarrow$  1-naphthol-6 : 8-disulphonic acid. 70% NaOH.Aq. at  $175^\circ \rightarrow$  1-amino-8-naphthol-4 : 6-disulphonic acid. Sulphonation with 25% anhydro-acid  $\rightarrow$  naphthasultam-2 : 4 : 6-trisulphonic acid.

Kalle, D.R.P., 275,449; 233,934, (*Chem. Zentr.*, 1914, II, 281; 1911, I, 1468).

**2-Naphthylamine-1 : 3 : 7-trisulphonic Acid** $C_{10}H_9O_3NS_3$ 

MW, 383

Boiled with dil. min. acids  $\rightarrow$  2-naphthylamine-3 : 7-disulphonic acid. Sulphonation with 40% anhydro-acid at  $130^\circ \rightarrow$  the 3 : 5 : 7- and 3 : 6 : 7-trisulphonic acids + 1 : 3 : 6 : 7-tetra-sulphonic acid. Di-Na salt : leaflets +  $4H_2O$ . Easily sol.  $H_2O$  with violet-blue fluor.

Dressel, Kothe, *Ber.*, 1894, 27, 1199.



## 2-Naphthylamine-1 : 5 : 7-trisulphonic Acid

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### 2-Naphthylamine-1 : 5 : 7-trisulphonic Acid.

Salts easily sol.  $\text{H}_2\text{O}$  with blue fluor. Does not couple with diazo-comps. The diazo-comp. +  $\text{Na}_2\text{CO}_3$ .Aq.  $\longrightarrow$  diazo-oxide of 2-amino-1-naphthol-5 : 7-disulphonic acid. 60%  $\text{NaOH}$ .Aq. at 160–220°  $\longrightarrow$  2-amino-5-naphthol-1 : 7-disulphonic acid. Part hyd. by boiling dil. min. acids  $\longrightarrow$  2-naphthylamine-5 : 7-disulphonic acid. Heat with 30% anhydro-acid at 140–60°  $\longrightarrow$  2-naphthylamine-3 : 5 : 7-trisulphonic acid.

Ofitzerov, *Chem. Abstracts*, 1935, 29, 1086.

### 2-Naphthylamine-3 : 5 : 7-trisulphonic Acid.

Salts very sol.  $\text{H}_2\text{O}$  with intense green fluor. 30%  $\text{NaOH}$ .Aq. at 190°  $\longrightarrow$  2-amino-5-naphthol-3 : 7-disulphonic acid. Tri-Na salt : needles +  $5\frac{1}{2}\text{H}_2\text{O}$  from EtOH.Aq.

Dressel, Kothe, *Ber.*, 1894, 27, 1202.

### 2-Naphthylamine-3 : 6 : 7-trisulphonic Acid.

Dil. aq. sols of salts show intense blue fluor. 55%  $\text{NaOH}$ .Aq. at 180–240°  $\longrightarrow$  2-amino-7-naphthol-3 : 6-disulphonic acid. Sulphonation with 40% anhydro-acid at 100–30°  $\longrightarrow$  2-naphthylamine-1 : 3 : 6 : 7-tetrasulphonic acid. Di-Na salt : needles +  $3\text{H}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Very spar. sol. cold  $\text{HCl}$ . Tri-Na salt : easily sol.  $\text{H}_2\text{O}$ .

See previous reference.

### 2-Naphthylamine-3 : 6 : 8-trisulphonic Acid.

Salts in aq. sol. show intense sky-blue fluor. 80%  $\text{NaOH}$ .Aq. at 220–60°  $\longrightarrow$  2-amino-8-naphthol-3 : 6-disulphonic acid. Zn dust + boiling  $\text{NaOH}$ .Aq.  $\longrightarrow$  2-naphthylamine-3 : 6-disulphonic acid. Di-K salt : needles +  $1\frac{1}{2}\text{H}_2\text{O}$ . Sol. 40 parts  $\text{H}_2\text{O}$  at 20°, 13 parts at 100°.

Kalle, D.R.P., 176,621, (*Chem. Zentr.*, 1906, II, 1746).

See also previous reference.

### 2-Naphthylamine-4 : 6 : 8-trisulphonic Acid.

60%  $\text{NaOH}$ .Aq. at 170–80°  $\longrightarrow$  2-amino-4-naphthol-6 : 8-disulphonic acid. Di-Na salt : micro-needles. Aq. sol.  $\longrightarrow$  brilliant blue fluor.

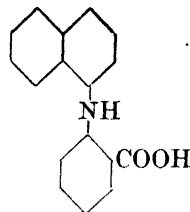
Morgan, Mitchell, *J. Chem. Soc.*, 1932, 1910.

### 1-Naphthylaminopropionic Acid.

See Naphthylalanine.

## 2-Naphthyl azide

**N- $\alpha$ -Naphthylanthranilic Acid** (2-[1-Naphthylamino-]-benzoic acid, N-phenyl-1-naphthylamine-2'-carboxylic acid, 2-carboxyphenyl- $\alpha$ -naphthylamine)

 $\text{C}_{17}\text{H}_{13}\text{O}_2\text{N}$ 

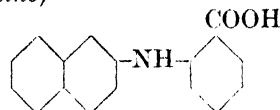
MW, 263

Leaflets or needles from  $\text{C}_6\text{H}_6$ -ligroin or EtOH. M.p. 208° (204–6°). Sol.  $\text{C}_6\text{H}_6$ , EtOH, AcOH. Spar. sol. hot  $\text{H}_2\text{O}$ , ligroin. Insol. cold  $\text{H}_2\text{O}$ . Sol. in conc.  $\text{H}_2\text{SO}_4$  on warming  $\longrightarrow$  yellow col. + bluish-green fluor.

Ullmann, Rasetti, *Ann.*, 1907, 355, 348.

Houben, Brassert, *Ber.*, 1906, 39, 3239.

**N- $\beta$ -Naphthylanthranilic Acid** (2-[2-Naphthylamino-]-benzoic acid, N-phenyl-2-naphthylamine-2'-carboxylic acid, 2-carboxyphenyl- $\beta$ -naphthylamine)

 $\text{C}_{17}\text{H}_{13}\text{O}_2\text{N}$ 

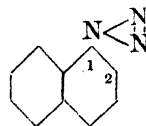
MW, 263

Needles from EtOH or  $\text{Me}_2\text{CO}$ . M.p. 212° (208–9°). Sol. hot  $\text{C}_6\text{H}_6$ , EtOH,  $\text{Me}_2\text{CO}$ . Very spar. sol. ligroin. Insol.  $\text{H}_2\text{O}$ .

Bucherer, Seyde, *J. prakt. Chem.*, 1907, 75, 279.

See also first reference above.

### 1-Naphthyl azide (1-Azidonaphthalene)

 $\text{C}_{10}\text{H}_7\text{N}_3$ 

MW, 169

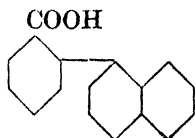
Prisms. M.p. 12°. Decomp. by heat or by conc.  $\text{H}_2\text{SO}_4$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ .

Forster, Fierz, *J. Chem. Soc.*, 1907, 91, 1945.

### 2-Naphthyl azide (2-Azidonaphthalene).

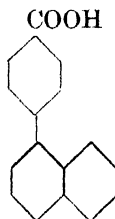
Needles or prisms. M.p. 33°. Sol. ord. org. solvents. Very spar. sol. boiling  $\text{H}_2\text{O}$ . Decomp. by 66%  $\text{H}_2\text{SO}_4$ . Turns yellow on exposure to light.

See previous reference.

**o-1-Naphthylbenzoic Acid** ( $\alpha$ -o-Carboxyphenylnaphthalene) $C_{17}H_{12}O_2$ 

MW, 248

Cryst. from AcOH. M.p. 229°.

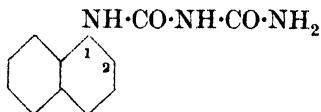
Nitrile:  $C_{17}H_{11}N$ . MW, 229. M.p. 73–7°. B.p. 230–40°/14 mm.Braun, Anton, *Ber.*, 1934, **67**, 1056.**p-1-Naphthylbenzoic Acid** ( $\alpha$ -p-Carboxyphenylnaphthalene) $C_{17}H_{12}O_2$ 

MW, 248

Rhombic cryst. from MeOH. M.p. 161·5°.

Schaarschmidt, Georgeacopol, *Ber.*, 1918, **51**, 1083.**o-2-Naphthylbenzoic Acid.**See  $\alpha$ -Chrysenic Acid.**Naphthylbenzylamine.**

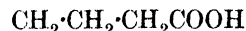
See Benzylnaphthylamine.

**1-Naphthylbiuret** ( $\alpha$ -Naphthylallophanamide) $C_{12}H_{11}O_2N_3$ 

MW, 229

Needles from EtOH.Aq. M.p. 259° (211°, 217°). Decomp. above m.p.  $\rightarrow$  1-naphthylurea. Spar. sol.  $H_2O$ ,  $Et_2O$ . Does not give biuret test.Bougault, Leboucq, *Bull. soc. chim.*, 1930, **47**, 600.Davis, Blanchard, *J. Am. Chem. Soc.*, 1929, **51**, 1805.**2-Naphthylbiuret** ( $\beta$ -Naphthylallophanamide).Needles. M.p. 230° (203°). Decomp. above m.p.  $\rightarrow$  2-naphthylurea.

See first reference above.

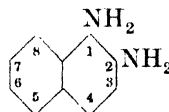
**3- $\alpha$ -Naphthylbutyric Acid** $C_{14}H_{14}O_2$ 

MW, 214

Cryst. M.p. 106–7°. B.p. 217°/15 mm.

Amide:  $C_{14}H_{15}ON$ . MW, 213. M.p. 160°.Willgerodt, *J. prakt. Chem.*, 1909, **80**, 183.  
Schroeter, Müller, Huang, *Ber.*, 1929, **62**, 656.**3- $\beta$ -Naphthylbutyric Acid.**

Leaflets from pet. ether or EtOH.Aq. M.p. 100° (94–5°).

Et ester:  $C_{16}H_{18}O_2$ . MW, 242. Oil. B.p. 216–18°/20 mm.Borsche, *Ber.*, 1919, **52**, 2083.Schroeter, Müller, Huang, *Ber.*, 1929, **62**, 657.**1:2-Naphthylenediamine** ( $\alpha$ : $\beta$ -Diaminonaphthalene) $C_{10}H_{10}N_2$ 

MW, 158

Leaflets from hot  $H_2O$ . M.p. 98°. B.p. 150–1°/0·5 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ .  $FeCl_3$  on HCl sol.  $\rightarrow$  green col. Forms add. comps.: with catechol, m.p. 104·5°; with sym.-trinitrobenzene, m.p. 203–4°.

1-N-Benzenesulphonyl: m.p. 215° decomp.

1:2-N-Diacetyl: needles. M.p. 234°.

1:2-N-Dibenzoyl: leaflets. M.p. 291°.

1-N-Phenyl:  $C_{16}H_{14}N_2$ . MW, 234. M.p. 170° (161°). Benzoyl deriv.: m.p. 215°.

2-N-Phenyl: m.p. 136–7° (138–40°). 1-N-Me: m.p. 85°.

Reilly, Drumm, O'Sullivan, *J. Soc. Chem. Ind.*, 1927, **46**, 437T.Fischer, Kracker, *J. prakt. Chem.*, 1922, **104**, 118.Erdmann, *Ber.*, 1903, **36**, 3461.Gattermann, Schulze, *Ber.*, 1897, **30**, 53.Bamberger, Schieffelin, *Ber.*, 1889, **22**, 1376.Harden, *Ann.*, 1889, **255**, 161.Noelting, Grandmougin, Freimann, *Ber.*, 1909, **42**, 1380.Zincke, Lawson, *Ber.*, 1887, **20**, 1170.Witt, *ibid.*, 573, 1184.

**1 : 3-Naphthylenediamine** (1 : 3-Diamino-naphthalene).

Plates from  $\text{H}_2\text{O}$ . M.p.  $96^\circ$ .

1 : 3-N-Diacetyl : needles from AcOH. M.p.  $263-5^\circ$ .

Atkinson, Thorpe, *J. Chem. Soc.*, 1906, **89**, 1922.

**1 : 4-Naphthylenediamine** (1 : 4-Diamino-naphthalene).

Bright yellow needles. M.p.  $120^\circ$ . Unstable in moist air. Forms add. comp. with *sym*-trinitrobenzene, m.p.  $208^\circ$  decomp.

1 : 4-N-Diacetyl : m.p.  $303-4^\circ$ .

1 : 4-N-Dibenzoyl : m.p.  $280.5^\circ$ .

1-N-p-Toluenesulphonyl : m.p.  $187-8^\circ$ . 1-N-Benzoyl : m.p.  $186^\circ$ .

1 : 4-N-Tetra-Et :  $\text{C}_{18}\text{H}_{26}\text{N}_2$ . MW, 270. M.p.  $47-8^\circ$ .

N-Phenyl : plates from  $\text{C}_6\text{H}_6$ . M.p.  $148^\circ$ . Diacetyl deriv. : needles from AcOH. M.p.  $278^\circ$ .

Kuhn, Wassermann, *Helv. Chim. Acta*, 1928, **11**, 79.

Krollpfeiffer, *Ann.*, 1923, **430**, 199.

Morgan, U.S.P., 1,442,818; E.P., 160,853, (*Chem. Abstracts*, 1923, **17**, 1242; 1921, **15**, 2445).

Cobenzyl, *Chem.-Ztg.*, 1915, **39**, 860.

Casale-Sacchi, *Gazz. chim. ital.*, 1914, **44**, ii, 398.

Fischer, *Ann.*, 1895, **286**, 183.

Weiss, Woidich, *Monatsh.*, 1925, **46**, 458.

**1 : 5-Naphthylenediamine** (1 : 5-Diamino-naphthalene).

Prisms from  $\text{H}_2\text{O}$ . M.p.  $190^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Mod. sol. hot.  $\text{H}_2\text{O}$ . Sublimes.  $\text{FeCl}_3$  on aq. suspension  $\rightarrow$  bluish-violet col. Forms add. comps. : with *m*-dinitrobenzene, m.p.  $78-9^\circ$ ; with *sym*-trinitrobenzene, m.p.  $245^\circ$ ; with 1 : 3 : 5-trinitronaphthalene, black needles, decomp. at  $243^\circ$ .

1 : 5-N-Diacetyl : m.p.  $360^\circ$ .

1 : 5-N-Tetra-Et : m.p.  $41^\circ$ .

Lenkhold, *Chem. Abstracts*, 1933, **27**, 5279.

Kunckell, Schneider, *Chem.-Ztg.*, 1912, **36**, 1021.

Bucherer, Ullmann, *J. prakt. Chem.*, 1909, **80**, 212.

Möller, *Elektrochemische Zeitschrift*, 1904, **10**, 201.

Meyer, Müller, *Ber.*, 1897, **30**, 774.

See also first and second references above.

**1 : 6-Naphthylenediamine** (1 : 6-Diamino-naphthalene).

Needles. M.p.  $85-6^\circ$  ( $77.5^\circ$ ). Sol. EtOH,  $\text{C}_6\text{H}_6$ , hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ , cold  $\text{H}_2\text{O}$ .  $\text{H}_2\text{O}$  sol. shows blue fluor.

1 : 6-N-Diacetyl : m.p.  $257^\circ$  ( $263.5^\circ$ ).

1 : 6-N-Dibenzoyl : m.p.  $265^\circ$ .

Krollpfeiffer, *Ann.*, 1923, **430**, 199.

Sachs, *Ber.*, 1906, **39**, 3022.

Bucherer, Wahl, *J. prakt. Chem.*, 1922, **103**, 253.

**1 : 7-Naphthylenediamine** (1 : 7-Diamino-naphthalene).

Leaflets from  $\text{C}_6\text{H}_6$ . M.p.  $117.5^\circ$ . Sol. EtOH,  $\text{C}_6\text{H}_6$ . Mod. sol. hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ , ligroin.

1 : 7-N-Diacetyl : m.p.  $213^\circ$ .

Friedländer *et al.*, *Ber.*, 1896, **29**, 41; 1892, **25**, 2082.

**1 : 8-Naphthylenediamine** (1 : 8-Diamino-naphthalene).

Needles from EtOH.Aq. M.p.  $66.5^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Sublimes.  $\text{FeCl}_3 \rightarrow$  brown ppt. Forms add. comp. with *sym*-trinitrobenzene, m.p.  $225^\circ$ .

1 : 8-N-Dibenzoyl : needles. M.p.  $311-12^\circ$ .

1 : 8-N-Tetra-Et : m.p.  $31-2^\circ$ .

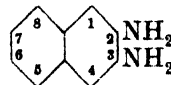
Kuhn, Wassermann, *Helv. Chim. Acta*, 1928, **11**, 79.

Krollpfeiffer, *Ann.*, 1923, **430**, 199.

Möller, *Elektrochemische Zeitschrift*, 1904, **10**, 222.

Ullmann, Consonno, *Ber.*, 1902, **35**, 2801.

Meyer, Müller, *Ber.*, 1897, **30**, 775.

**2 : 3-Naphthylenediamine** (2 : 3-Diamino-naphthalene)

$\text{C}_{10}\text{H}_{10}\text{N}_2$  MW, 158

Leaflets from  $\text{H}_2\text{O}$ . M.p.  $196^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ .

2 : 3-N-Diacetyl : m.p.  $247^\circ$ .

2 : 3-N-Tetra-Et :  $\text{C}_{18}\text{H}_{26}\text{N}_2$ . MW, 270. M.p.  $75-6^\circ$ .

Morgan, Godden, *J. Chem. Soc.*, 1910, **97**, 1718.

Sachs, *Ber.*, 1906, **39**, 3021.

See also first two references above.

**2 : 6-Naphthylenediamine** (2 : 6-Diamino-naphthalene).

Needles or leaflets from  $\text{H}_2\text{O}$ . M.p.  $216-17^\circ$ . Spar. sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ .

## 2 : 7-Naphthylenediamine

48

2 : 6-N-Tetra-Et : m.p. 69–70°.

Kuhn, Wassermann, *Helv. Chim. Acta*, 1928, 11, 79.

Krollpfeiffer, *Ann.*, 1923, 430, 199.

Jacchia, *Ann.*, 1902, 323, 132.

Friedländer, Lucht, *Ber.*, 1893, 26, 3033.

**2 : 7-Naphthylenediamine** (2 : 7-Diamino-naphthalene).

Leaflets from H<sub>2</sub>O. M.p. 166° (159°).

2 : 7-N-Diacetyl : m.p. 261°.

2 : 7-N-Dibenzoyl : m.p. 267°.

2 : 7-N-Tetra-Et : b.p. 234–6°/13 mm.

Dipicrate : m.p. 210°.

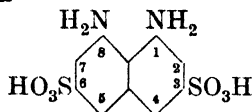
Kuhn, Jacob, Furter, *Ann.*, 1927, 455, 270.

Windaus, *Ber.*, 1924, 57, 1737.

Kaufler, Karrer, *Ber.*, 1907, 40, 3262.

See also first two references above.

**1 : 8-Naphthylenediamine - 3 : 6-disulphonic Acid**



C<sub>10</sub>H<sub>10</sub>O<sub>6</sub>N<sub>2</sub>S<sub>2</sub> MW, 318

Sols. of salts fluor. green. Heat with dil. acids at 100–20°, or with NaOH.Aq. at 200–10° → 8-amino-1-naphthol-3 : 6-disulphonic acid. Heat with dil. acids at 150–60° or with NaOH.Aq. at 260–80° → 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid. Condenses with 1 mol. Me<sub>2</sub>CO in presence of min. acids. Mono-K salt : needles + 3H<sub>2</sub>O. Sol. hot H<sub>2</sub>O. Ba salt : needles + 6H<sub>2</sub>O. Sol. hot. H<sub>2</sub>O.

Alén, *Ber.*, 1884, 17 (Referate), 437.

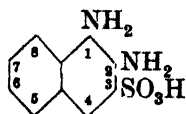
Badische, D.R.P., 121,228, (*Chem. Zentr.*, 1901, I, 1395); D.R.P., 122,475, (*Chem. Zentr.*, 1901, II, 447).

**1 : 8-Naphthylenediamine - 4 : 5-disulphonic Acid.**

Leaflets. Sol. H<sub>2</sub>O. Couples rapidly with 1 mol., less rapidly with 2 mols. diazo-comps. HNO<sub>2</sub> → azimino-comp. Heat with min. acids → 1-amino-8-naphthol-4 : 5-disulphonic acid. Na salt : sol. H<sub>2</sub>O.

Bucherer, Barsch, *J. prakt. Chem.*, 1925, 111, 313, 336.

**1 : 2-Naphthylenediamine - 3-sulphonic Acid**



C<sub>10</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>S

MW, 238

**1 : 3-Naphthylenediamine-6-sulphonic Acid**

Needles. Aq. sol. + FeCl<sub>3</sub> → intense emerald-green col. NaHg → 1 : 2-naphthylenediamine.

Gattermann, Liebermann, *Ann.*, 1912, 393, 209.

**1 : 2-Naphthylenediamine - 4-sulphonic Acid.**

Needles from hot H<sub>2</sub>O. NaHg → 1 : 2-naphthylenediamine.

Friedländer, Kielbasinski, *Ber.*, 1896, 29, 1978.

**1 : 2-Naphthylenediamine - 5-sulphonic Acid.**

Light brown glittering leaflets. Spar. sol. H<sub>2</sub>O. Aq. sol. + FeCl<sub>3</sub> → emerald-green col. NaHg → 1 : 2-naphthylenediamine.

Gattermann, Liebermann, *Ann.*, 1912, 393, 205.

**1 : 2-Naphthylenediamine - 6-sulphonic Acid.**

Needles. Very spar. sol. pure H<sub>2</sub>O. Aq. sol. + FeCl<sub>3</sub> → dirty green ppt. With Ac<sub>2</sub>O → an iminazole. With phenanthraquinone → an azine.

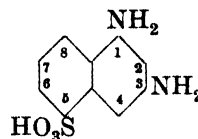
Witt, *Ber.*, 1888, 21, 3484.

**1 : 2-Naphthylenediamine - 7-sulphonic Acid.**

Grey powder. Spar. sol. H<sub>2</sub>O. Aq. sol. + FeCl<sub>3</sub> → dirty green ppt.

See previous reference.

**1 : 3-Naphthylenediamine - 5-sulphonic Acid**



C<sub>10</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>S

MW, 238

Needles. Sol. H<sub>2</sub>O.

Dannerth, *J. Am. Chem. Soc.*, 1907, 29, 1327.

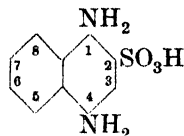
**1 : 3-Naphthylenediamine - 6-sulphonic Acid.**

Needles. Spar. sol. cold H<sub>2</sub>O. Alkali salts easily sol. H<sub>2</sub>O.

Friedländer, Taussig, *Ber.*, 1897, 30, 1462.

**1 : 4-Naphthylenediamine-2-sulphonic Acid**

**1 : 4 - Naphthylenediamine - 2 - sulphonic Acid**



$C_{10}H_{10}O_3N_2S$  MW, 238

Reddish needles. Very spar. sol. boiling  $H_2O$ . The yellow sol. in  $AcONa.Aq.$  shows green fluor.  $NaHg \rightarrow$  1 : 4-naphthylenediamine.

Gattermann, Liebermann, *Ann.*, 1912, 393, 209.

**1 : 4 - Naphthylenediamine - 5 - sulphonic Acid.**

Steel-blue leaflets.  $NaHg \rightarrow$  1 : 4-naphthylenediamine.

See previous reference.

**1 : 4 - Naphthylenediamine - 6 - sulphonic Acid.**

Needles. Stable when dry. Sol. 223 parts  $H_2O$ . Sol. inorganic acids.

1-N-Acetyl: needles. Very spar. sol. hot  $H_2O$ .

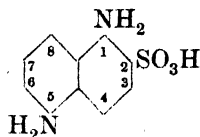
4-N-Acetyl: needles. Spar. sol.  $H_2O$ . Aq. sol. +  $FeCl_3$  or  $CrO_3 \rightarrow$  blue col. The product of ox. by alk.  $KMnO_4$  when fused with  $KOH$  at  $200-20^\circ \rightarrow$  4-hydroxyphthalic acid.

Fabriowicz, Lesnianski, *Chem. Abstracts*, 1932, 26, 3791.

Gaess, D.R.P., 138,030, (*Chem. Zentr.*, 1903, I, 109).

Ammelburg, *J. prakt. Chem.*, 1893, 48, 286.

**1 : 5 - Naphthylenediamine - 2 - sulphonic Acid**



$C_{10}H_{10}O_3N_2S$  MW, 238

Cryst. +  $3H_2O$ . Spar. sol.  $H_2O$ . Heat with dil. min. acids  $\rightarrow$  1 : 5-naphthylenediamine.

Hydrochloride: spar. sol. cold  $H_2O$ .

Sulphate: insol.  $H_2O$ .

Bucherer, Ullmann, *J. prakt. Chem.*, 1909, 80, 213.

Friedländer, Kielbasinski, *Ber.*, 1896, 29, 1983.

**1 : 5 - Naphthylenediamine - 4 - sulphonic Acid.**

Bluish-grey leaflets.

44

**2-β-Naphthylethyl Alcohol**

N-Acetyl: spar. sol.  $H_2O$ .

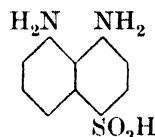
See first reference above.

**1 : 6 - Naphthylenediamine - 4 - sulphonic Acid.**

Needles from hot  $H_2O$ . Very spar. sol. cold  $H_2O$ . Sols. of salts fluor. strongly bluish-violet.

Friedländer, Kielbasinski, *Ber.*, 1896, 29, 1979.

**1 : 8 - Naphthylenediamine - 4 - sulphonic Acid**



$C_{10}H_{10}O_3N_2S$  MW, 238

Leaflets. Very spar. sol.  $H_2O$ . The alkali salts are very sol.  $H_2O$ . Couples with 1 mol. or 2 mols. diazo-comps.  $HNO_2 \rightarrow$  azimino-comp. Na salt heated with  $Na_2SO_3 \rightarrow$  8-amino-1-naphthol-4-sulphonic acid. Heat with milk of lime under press. at  $220-40^\circ \rightarrow$  1 : 8-dihydroxynaphthalene-4-sulphonic acid.

Bayer, D.R.P., 216,075, (*Chem. Zentr.*, 1909, II, 1950).

Badische, D.R.P., 120,690, (*Chem. Zentr.*, 1901, I, 1395).

**Naphthylenediphenyldiamine.**

See Diphenylnaphthylenediamine.

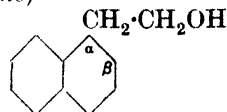
**Naphthyleneditolyldiamine.**

See Ditolylnaphthylenediamine.

**Naphthylethane.**

See Ethylnaphthalene.

**2-α-Naphthylethyl Alcohol (1-β-Hydroxyethylnaphthalene)**



$C_{12}H_{12}O$  MW, 172

Cryst. M.p.  $62^\circ$ . B.p.  $174-8^\circ/13\text{ mm.}$ ,  $125^\circ/0.2\text{ mm.}$  Heat with  $KOH$  under reduced press.  $\rightarrow$  1-naphthylethylene.

Haworth, Mavin, *J. Chem. Soc.*, 1933, 1014.

Sontag, *Ann. chim.*, 1934, 1, 399.

Shorugin, Shorugin, *Chem. Abstracts*, 1935, 29, 6886.

**2-β-Naphthylethyl Alcohol (2-β-Hydroxyethylnaphthalene).**

Needles from ligroin. M.p.  $68^\circ$ . B.p.  $178-88^\circ/15\text{ mm.}$  Sol.  $Et_2O$ ,  $EtOH$ . Orange-like

odour. Heat with KOH  $\longrightarrow$  2-naphthylethyl-ene.

See second reference above.

**1- $\alpha$ -Naphthylethylamine** (1- $\alpha$ -Aminoethyl-naphthalene, 1-methyl- $\alpha$ -naphthylmethylaniline)



$C_{12}H_{13}N$

MW, 171

*d*l-.

B.p.  $153^\circ/11$  mm.  $D_{25}^{25}$  1.055.  $[\alpha]_D^{17} + 82.8^\circ$ ,  $[\alpha]_D^{19} + 61.6^\circ$  in EtOH.

*B*, *HCl*: cryst. +  $H_2O$ .  $[\alpha]_D^{18} + 3.9^\circ$  in  $H_2O$ .

*l*l-.

Liq.  $[\alpha]_D^{25} - 80.8^\circ$ ,  $[\alpha]_D^{25} - 60.8^\circ$  in EtOH.

*B*, *HCl*:  $[\alpha]_D^{18} - 3.9^\circ$  in  $H_2O$ .

$B_2H_2SO_4$ : needles +  $4H_2O$ . M.p.  $230-2^\circ$ .

Oxalate: prisms. M.p.  $232^\circ$  decomp.

$\alpha$ -Camphorate: needles. M.p.  $212-13^\circ$  decomp.

Acid- $\alpha$ -camphorate: needles. M.p.  $196^\circ$ .

Acid-d-tartrate: needles +  $H_2O$ . M.p.  $186^\circ$ .

Urea deriv.: m.p.  $186^\circ$ .

*dl*l-.

Liq. B.p.  $156^\circ/15$  mm.,  $153^\circ/11$  mm.

*B*, *HCl*: needles or prisms. Decomp. at  $240-5^\circ$ .

$B_2H_2SO_4$ : cryst. +  $H_2O$ . M.p.  $233^\circ$ .

Oxalate: needles. M.p.  $221^\circ$  decomp.

Urea deriv.: m.p.  $181-2^\circ$ .

Benzoyl: m.p.  $166-166.5^\circ$ .

Picrate: m.p.  $212-13^\circ$  decomp.

Samuelsson, *Chem. Abstracts*, 1924, **18**, 1833.

**1- $\beta$ -Naphthylethylamine** (2- $\alpha$ -Aminoethyl-naphthalene, 1-methyl- $\beta$ -naphthylmethylaniline).

*d*l-.

M.p.  $53^\circ$ .  $[\alpha]_D^{19} + 19.4^\circ$  in EtOH.

*B*, *HCl*: needles. M.p.  $219^\circ$ .  $[\alpha]_D^{17} + 5.4^\circ$  in  $H_2O$ .

$B_2H_2SO_4$ : needles +  $2H_2O$ . M.p.  $262-3^\circ$ .

Oxalate: m.p.  $240^\circ$ .

Acid-d-tartrate: needles +  $H_2O$ . M.p.  $199-200^\circ$ .

Urea deriv.: m.p.  $182^\circ$ .  $[\alpha]_D^{17} + 67.9^\circ$  in EtOH.

*l*l-.

M.p.  $53^\circ$ .  $[\alpha]_D^{20} - 18.9^\circ$  in EtOH.

*B*, *HCl*: m.p.  $219^\circ$ .  $[\alpha]_D^{17} - 6.0^\circ$  in  $H_2O$ .

Urea deriv.: m.p.  $182^\circ$ .  $[\alpha]_D^{17} - 66.8^\circ$  in EtOH.

*dl*l-.

M.p.  $23^\circ$ . B.p.  $142-3^\circ/6-7$  mm.  $D_{20}^{20}$  1.047.

*B*, *HCl*: needles. M.p.  $199-200^\circ$ .

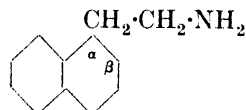
$B_2H_2SO_4$ : needles. M.p.  $243-4^\circ$ .

Oxalate: needles. M.p.  $232-3^\circ$ .

Urea deriv.: m.p.  $198^\circ$ .

See previous reference.

**2- $\alpha$ -Naphthylethylamine** (1- $\beta$ -Aminoethyl-naphthalene)



$C_{12}H_{13}N$

MW, 171

B.p.  $182-3^\circ/18$  mm. ( $170-3^\circ/16$  mm.).

*B*, *HCl*: m.p.  $243-8^\circ$ .

*N*-Acetyl: m.p.  $91^\circ$ .

*N*-Benzoyl: needles from ligroin. M.p.  $97^\circ$  (softens at  $87^\circ$ ).

Picrate: m.p.  $201-2^\circ$ .

Mayer *et al.*, *Ber.*, 1923, **56**, 1413; 1922, **55**, 1847.

**2- $\beta$ -Naphthylethylamine** (2- $\beta$ -Aminoethyl-naphthalene).

Cryst. below room temp. B.p.  $174-5^\circ/25$  mm. ( $160-5^\circ/15$  mm.).

*B*, *HCl*: m.p.  $250^\circ$  decomp.

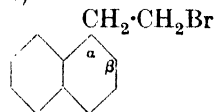
*N*-Acetyl: m.p.  $110^\circ$ .

*N*-Benzoyl: prisms from  $C_6H_6$ . M.p.  $140-1^\circ$ .

Picrate: m.p.  $196^\circ$ .

Mayer *et al.*, *Ber.*, 1923, **56**, 1411; 1922, **55**, 1858.

**2- $\alpha$ -Naphthylethyl bromide** (1- $\beta$ -Bromoethyl-naphthalene)



$C_{12}H_{11}Br$

MW, 235

B.p.  $172^\circ/20$  mm.,  $145-8^\circ/0.3$  mm.,  $114^\circ/0.15$  mm.

Kon, *J. Chem. Soc.*, 1933, 1085.

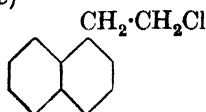
Ruzicka, Bossard, Schmid, *Helv. Chim. Acta*, 1933, **16**, 836.

Haworth, Mavin, *J. Chem. Soc.*, 1933, 1014.

**2- $\beta$ -Naphthylethyl bromide** (2- $\beta$ -Bromoethyl-naphthalene).

M.p.  $59^\circ$ . B.p.  $146-7^\circ/18$  mm.

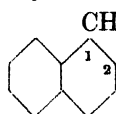
Sontag, *Ann. chim.*, 1934, **1**, 405.

**2- $\alpha$ -Naphthylethyl chloride** (1- $\beta$ -Chloro-ethylnaphthalene) $C_{12}H_{11}Cl$ 

MW, 190.5

Yellowish liq. B.p. 167-8°/17 mm.

Picrate : m.p. 67-8°.

Cook, Hewett, *J. Chem. Soc.*, 1933, 1107.**1-Naphthylethylene** (1-Vinylnaphthalene) $C_{12}H_{10}$ 

MW, 154

B.p. 124-5°/15 mm., 115-16°/3-4 mm.  $D_4^{18}$ 1.036.  $n_D^{20}$  1.644. Readily polymerises.

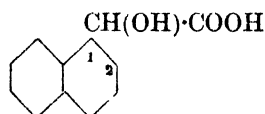
Picrate : m.p. 101-2° decomp.

Styphnate : m.p. 96-7°.

Sontag, *Ann. chim.*, 1934, 1, 400.I.G., U.S.P., 1,985,844, (*Chem. Abstracts*, 1935, 29, 1099).Shorugin, Shorugina, *Chem. Abstracts*, 1935, 29, 6866.**2-Naphthylethylene** (2-Vinylnaphthalene).

Cryst. from EtOH.Aq. M.p. 66°. B.p. 135-7°/18 mm. Intense thyme-like odour. More stable than the 1-isomer.

See first and second references above.

**1-Naphthylglycollic Acid** ( $\alpha$ -Naphthylglycollic acid) $C_{12}H_{10}O_3$ 

MW, 202

d-.

Leaflets from  $C_6H_6$ . M.p. 124-5°.  $[\alpha]_{5461}^{13} + 230^\circ$  in EtOH. Conc.  $H_2SO_4 \rightarrow$  blue col.Me ester :  $C_{13}H_{12}O_3$ . MW, 216. Liq. B.p. 230-2°/36 mm.  $[\alpha]_{5461}^{16} + 175^\circ$  in  $Me_2CO$ .l-Menthyl ester : cryst. from pet. ether. M.p. 71-2°.  $[\alpha]_{5461}^{20} + 27.6^\circ$  in  $Me_2CO$ .

l-.

Leaflets from  $C_6H_6$ . M.p. 124-5°.  $[\alpha]_{5461}^{16} - 228^\circ$  in EtOH.l-Menthyl ester : needles from pet. ether. M.p. 62-3°. B.p. 220°/24 mm.  $[\alpha]_{5461}^{20} - 148.7^\circ$  in  $Me_2CO$ .

dl-.

Needles from  $H_2O$ . M.p. 98-9° (91°). Sol.  $C_{10}H_{10}N_2$ EtOH, Et<sub>2</sub>O. Mod. sol.  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  blue col.

Me ester : needles from pet. ether. M.p. 79°.

Et ester :  $C_{14}H_{14}O_3$ . MW, 230. Needles from pet. ether. M.p. 69°.l-Menthyl ester : m.p. 38.5-39.5°.  $[\alpha]_{5461}^{21} - 61.4^\circ$  in  $Me_2CO$ .Amide :  $C_{12}H_{11}O_2N$ . MW, 201. Plates from  $Me_2CO$ -pet. ether. M.p. 134-5°.McKenzie, Dennler, *J. Chem. Soc.*, 1926, 1599; *Ber.*, 1927, 60, 221.McKenzie, Gow, *J. Chem. Soc.*, 1933, 35.Roger, Gow, *J. Chem. Soc.*, 1934, 130.**2-Naphthylglycollic Acid** ( $\beta$ -Naphthylglycollic acid).Cryst. from ligroin. M.p. 158°. Sol. hot EtOH, AcOH. Less sol. hot  $H_2O$ . Spar. sol. Et<sub>2</sub>O, ligroin.

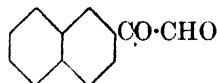
Me ester : needles from EtOH. M.p. 75°.

Et ester : needles from ligroin. M.p. 87°.

Amide : leaflets from EtOH. M.p. 227-8°.

Nitrile : benzoyl : cryst. from  $PhNO_2$ . M.p. 239°.

Acetyl : cryst. from AcOH. M.p. 150°.

Schweitzer, *Ber.*, 1891, 24, 549.Madelung, Oberwegner, *Ber.*, 1932, 65, 940. **$\beta$ -Naphthylglyoxal** $C_{12}H_8O_2$ 

MW, 184

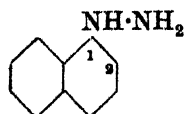
Intensely yellow oil. B.p. 183°/20 mm. part decomp.

Diacetate :  $C_{10}H_7 \cdot CO \cdot CH(O \cdot CO \cdot CH_3)_2$ . Prisms from  $C_6H_6$ -pet. ether. M.p. 150°.Di-Me acetal :  $C_{10}H_7 \cdot CO \cdot CH(O \cdot CH_3)_2$ . B.p. 194°/16 mm.Hydrate :  $C_{10}H_7 \cdot CO \cdot CH(OH)_2$ . Leaflets from  $H_2O$ . M.p. 98°.Madelung, Oberwegner, *Ber.*, 1932, 65, 939.**Naphthylglyoxylic Acid.**

See Naphthoylformic Acid.

**N-Naphthylheptadecylamine.**

See Heptadecylnaphthylamine.

**1-Naphthylhydrazine**

MW, 158

Plates from  $\text{H}_2\text{O}$ . M.p. 116–17°. B.p. 203°/20 mm. Very sol. hot  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Mild ox. agents give naphthalene +  $\text{N}_2$ .

*Acetyl deriv.*: needles from  $\text{EtOH.Aq}$ . M.p. 143°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*N'-Phenyl*: leaflets. M.p. 124°. N : N'-*Di-acetyl*: m.p. 264°.

*N'-o-Tolyl*: m.p. 107°. N : N'-*Diacetyl*: m.p. 252°.

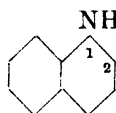
Fischer, *Ann.*, 1886, 232, 237.

### 2-Naphthylhydrazine.

Leaflets from  $\text{H}_2\text{O}$ . M.p. 124–5°. Sol. hot  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ .  $\text{FeCl}_3 \rightarrow$  naphthalene +  $\text{N}_2$ .  $\text{HCl}$  in sealed tube at 200°  $\rightarrow$  2-naphthylamine +  $\text{NH}_3$ .

See previous reference.

### 1-Naphthylhydroxylamine



$\text{C}_{10}\text{H}_9\text{ON}$

MW, 159

Cryst. +  $\text{H}_2\text{O}$ . M.p. anhyd. 79°. Sol. org. solvents. Mod. sol.  $\text{H}_2\text{O}$ . Reduces  $\text{NH}_3\text{.AgNO}_3$  and Fehling's.  $\text{Ag}_2\text{O} \rightarrow$  1-nitrosonaphthalene.

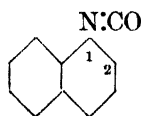
Willstätter, Kubli, *Ber.*, 1908, 41, 1937.

### 2-Naphthylhydroxylamine.

Leaflets from  $\text{CHCl}_3$ . M.p. 126°.

Baudisch, Furst, *Ber.*, 1917, 50, 324.

### 1-Naphthyl isocyanate



$\text{C}_{11}\text{H}_7\text{ON}$

MW, 169

Liq. B.p. 269–70°.

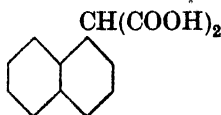
Vittenet, *Bull. soc. chim.*, 1899, 21, 957.

### 2-Naphthyl isocyanate.

Leaflets. M.p. 55–6°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

See previous reference.

### 1-Naphthylmalonic Acid



$\text{C}_{13}\text{H}_{10}\text{O}_4$

MW, 230

*Di-Me ester*:  $\text{C}_{15}\text{H}_{14}\text{O}_4$ . MW, 258. Needles from 50%  $\text{EtOH}$ . M.p. 104°.

*Di-Et ester*:  $\text{C}_{17}\text{H}_{18}\text{O}_4$ . MW, 286. Prisms or plates from pet. ether. M.p. 59–60°. Very sol.  $\text{CHCl}_3$ . Sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ .

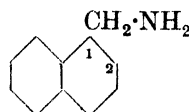
Keach, *J. Am. Chem. Soc.*, 1933, 55, 3440.

Wislicenus, Butterfass, Koken, *Ann.*, 1924, 436, 81.

### Naphthyl Mercaptan.

See Thionaphthol.

**1-Naphthylmethylamine** ( $\alpha$ -Menaphthylamine, 1- $\alpha$ -aminomethylnaphthalene)



$\text{C}_{11}\text{H}_{10}\text{N}$

MW, 156

Liq. B.p. 155°/12 mm.

*B.HCl*: m.p. 262–4°. Mod. sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*Acetyl*: needles from ligroin. M.p. 134°.

*Benzenesulphonyl*: m.p. 148°. Spar. sol. cold  $\text{EtOH}$ .

*Picrate*: m.p. 223°. Mod. sol.  $\text{EtOH}$ .

*Methiodide*: leaflets from  $\text{EtOH}$ . M.p. 213°.

*Phenylurea deriv.*: m.p. 216°. Spar. sol.  $\text{EtOH}$ .

v. Braun, Blessing, Zobel, *Ber.*, 1923, 56, 1996.

I.G., U.S.P., 1,873,402, (*Chem. Abstracts*, 1932, 26, 5965).

**2-Naphthylmethylamine** ( $\beta$ -Menaphthylamine, 2- $\alpha$ -aminomethylnaphthalene).

Cryst. M.p. 60°. B.p. 148–9°/12 mm.

*B.HCl*: m.p. 269°.

*Acetyl*: m.p. 126°.

*Picrate*: m.p. 226°. Spar. sol.  $\text{EtOH}$ .

*Methiodide*: prisms from  $\text{EtOH}$ . M.p. 168°.

See first reference above.

### 2- $\beta$ -Naphthyl-1-naphthoic Acid.

See Picenic Acid.

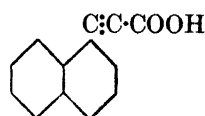
### Naphthylnitromethane.

See  $\omega$ -Nitromethylnaphthalene.

### Naphthylphenylenediamine.

See under Phenylenediamine.

### 1-Naphthylpropionic Acid



$\text{C}_{13}\text{H}_{10}\text{O}_2$

MW, 196

Needles from  $\text{H}_2\text{O}$  or  $\text{CS}_2$ . M.p. 138–9° decomp. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ . Spar. sol.  $\text{H}_2\text{O}$ ,



## 1- $\alpha$ -Naphthylpropionaldehyde

48

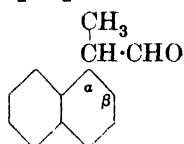
## 1-Naphthylsuccinic Acid

cold  $\text{CCl}_4$ . Turns yellow in air.  $\text{H}_2\text{O}$  is sealed tube at  $125^\circ \rightarrow$  1-naphthylacetylene.

Leroy, *Bull. soc. chim.*, 1892, 7, 645.

West, *J. Am. Chem. Soc.*, 1920, 42, 1666.

### 1- $\alpha$ -Naphthylpropionaldehyde



$\text{C}_{13}\text{H}_{12}\text{O}$  MW, 184

Liq. B.p.  $170^\circ/14$  mm.,  $131-2^\circ/4$  mm.  $D_4^{20}$  1.118.

Semicarbazone: m.p.  $209-10^\circ$ .

Darzens, *Compt. rend.*, 1907, 145, 1342.

Tiffeneau, Daudel, *Compt. rend.*, 1908, 147, 679.

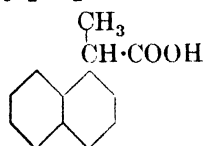
### 1- $\beta$ -Naphthylpropionaldehyde.

Cryst. M.p.  $53^\circ$ .

Semicarbazone: m.p.  $134-5^\circ$ .

See first reference above.

### 1- $\alpha$ -Naphthylpropionic Acid

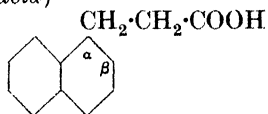


$\text{C}_{13}\text{H}_{12}\text{O}_2$  MW, 200

Cryst. M.p.  $145^\circ$ .

Tiffeneau, Daudel, *Compt. rend.*, 1908, 147, 679.

### 2- $\alpha$ -Naphthylpropionic Acid ( $\alpha$ -Mena-phthylacetic acid)



$\text{C}_{13}\text{H}_{12}\text{O}_2$  MW, 200

Needles from EtOH. M.p.  $156^\circ$  ( $148^\circ$ ). B.p.  $179^\circ/11$  mm.

Me ester:  $\text{C}_{14}\text{H}_{14}\text{O}_2$ . MW, 214. B.p.  $162^\circ/2$  mm.

Chloride:  $\text{C}_{13}\text{H}_{11}\text{OCl}$ . MW, 218.5. M.p.  $26^\circ$ . B.p.  $179-80^\circ/12$  mm.

Amide:  $\text{C}_{13}\text{H}_{13}\text{ON}$ . MW, 199. Needles from  $\text{H}_2\text{O}$ . M.p.  $140^\circ$  ( $133^\circ$ ).

Hydrazide: needles from EtOH. M.p.  $125-6^\circ$ .

Brandis, *Ber.*, 1889, 22, 2156.

Willgerodt, *J. prakt. Chem.*, 1909, 80, 183.

Darzens, Lévy, *Compt. rend.*, 1935, 201, 902.

### 2- $\beta$ -Naphthylpropionic Acid ( $\beta$ -Mena-phthylacetic acid).

Leaflets from  $\text{H}_2\text{O}$ . M.p.  $135^\circ$  ( $129-30^\circ$ ).

Et ester:  $\text{C}_{15}\text{H}_{16}\text{O}_2$ . MW, 228. Leaflets. M.p.  $28^\circ$ . B.p.  $195-8^\circ/25$  mm.

Amide: leaflets from  $\text{H}_2\text{O}$ . M.p.  $168^\circ$ .

Hydrazide: needles. M.p.  $156^\circ$ .

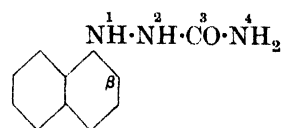
See last reference above and also

Monier-Williams, *J. Chem. Soc.*, 1906, 89, 277.

Willgerodt, *J. prakt. Chem.*, 1909, 80, 188.

Mayer, Schnecko, *Ber.*, 1923, 56, 1411.

### 1- $\alpha$ -Naphthylsemicarbazide



$\text{C}_{11}\text{H}_{11}\text{ON}_3$  MW, 201

Leaflets from hot amyl alcohol. M.p.  $231^\circ$ .

Spar. sol. cold EtOH. Insol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ .

Pinner, *Ber.*, 1888, 21, 1222.

### 1- $\beta$ -Naphthylsemicarbazide.

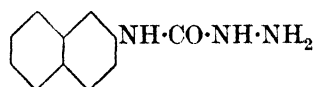
Leaflets from  $\text{H}_2\text{O}$ . M.p.  $225^\circ$ . Sol. hot EtOH, AcOH. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ . Insol. cold  $\text{H}_2\text{O}$ . Reduces warm Fehling's.

4-o-Tolyl: needles from AcOH. M.p.  $215^\circ$ . Spar. sol. EtOH.

4-p-Tolyl: needles from EtOH. M.p.  $187^\circ$ .

Pinner, *Ber.*, 1888, 21, 1223.

### 4- $\beta$ -Naphthylsemicarbazide



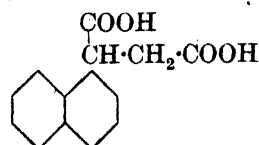
$\text{C}_{11}\text{H}_{11}\text{ON}_3$  MW, 201

Cryst. powder. M.p.  $258-9^\circ$ . Stable.

B, HCl: needles from  $\text{H}_2\text{O}$ . M.p. about  $260^\circ$  decomp.

Borsche, *Ber.*, 1901, 34, 4302; 1905, 38, 836.

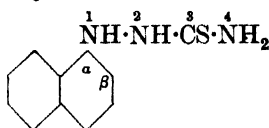
### 1-Naphthylsuccinic Acid



$\text{C}_{14}\text{H}_{12}\text{O}_4$  MW, 244

Leaflets from hot  $\text{H}_2\text{O}$ . M.p.  $206^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH. Spar. sol. pet. ether.

Wislicenus, Butterfass, Koken, *Ann.*, 1924, 436, 81.

1- $\alpha$ -Naphthylthiosemicarbazide $C_{11}H_{11}N_3S$ 

MW, 217

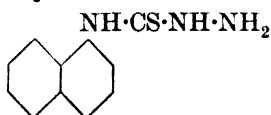
Leaflets from EtOH. M.p. 209° decomp.  
Spar. sol. EtOH,  $C_6H_6$ ,  $CS_2$ . Insol.  $Et_2O$ ,  $CHCl_3$ .

4-*Me*: m.p. 195°.4-*Et*: m.p. 149°.4-*p-Tolyl*: m.p. 169°.4- $\alpha$ -Naphthyl: m.p. 192°.4- $\beta$ -Naphthyl: m.p. 179°.Freund, Schuftan, *Ber.*, 1891, 24, 4190.Marckwald, *Ber.*, 1899, 32, 1087.1- $\beta$ -Naphthylthiosemicarbazide.

Cryst. from EtOH. M.p. 204° (201-2°). Sol.  
hot EtOH, hot aniline. Spar. sol.  $C_6H_6$ ,  $CS_2$ ,  
ligroin. Insol.  $H_2O$ ,  $Et_2O$ .

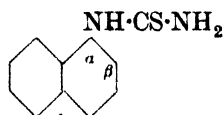
4-*Me*: m.p. 212° (209°).4-*Et*: m.p. 169°.4-*Allyl*: m.p. 155°.4-*Phenyl*: leaflets from EtOH. M.p. 202°.4-*o-Tolyl*: m.p. 192°.4-*p-Tolyl*: m.p. 195°.4- $\alpha$ -Naphthyl: m.p. 207°.4- $\beta$ -Naphthyl: m.p. 187°.

See last reference above and also

Hauff, *Ann.*, 1889, 253, 30.4- $\alpha$ -Naphthylthiosemicarbazide $C_{11}H_{11}N_3S$ 

MW, 217

Needles from EtOH, leaflets from  $C_6H_6$ .  
M.p. 138-9°. Mod. sol. most solvents. Spar.  
sol. ligroin.

Busch, Ulmer, *Ber.*, 1902, 35, 1715. $\alpha$ -Naphthylthiourea $C_{11}H_{10}N_2S$ 

MW, 202

Prisms from EtOH. M.p. 198°. Spar. sol.  
 $H_2O$ , cold EtOH,  $Et_2O$ .

N'-*Me*: m.p. 196°.N'-*Allyl*: m.p. 145°.

N'-*Phenyl*: plates. M.p. 162-3°. Spar. sol.  
EtOH,  $Et_2O$ . N'-*Me*: m.p. 135-6°. N'-*Et*:  
m.p. 129-129.5°.

Dict. of Org. Comp.—III.

N'-*o-Tolyl*: needles from EtOH. M.p. 167°.N'-*p-Tolyl*: needles from EtOH. M.p. 168°.N'-*Benzyl*: m.p. 172-3°.N'-Naphthyl: *see sym.*-Di-1-naphthylthiourea.N'-*Acetyl*: needles from EtOH. M.p. 198°.Sol. 40 parts boiling EtOH. Spar. sol.  $Et_2O$ .

N'-*Benzoyl*: yellow prisms from EtOH. M.p.  
172-3°. Sol. 50 parts hot EtOH.

Heller, Bauer, *J. prakt. Chem.*, 1902, 65, 380.de Clermont, Wehrlin, *Bull. soc. chim.*, 1876, 26, 126.Dyson, Hunter, *Chem. News*, 1927, 134, 4. $\beta$ -Naphthylthiourea.

Leaflets from EtOH. M.p. 186° (180°). Spar.  
sol. usual solvents.

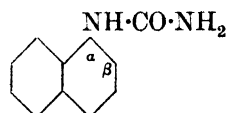
N'-*Phenyl*: leaflets from EtOH. M.p. 182-3°  
(165°). N'-*Me*: m.p. 124.5-125°. N'-*Et*: plates.  
M.p. 128.5-129°.

N'-*o-Tolyl*: m.p. 193-4°.N'-*p-Tolyl*: m.p. 163-4°.

N'-*Benzyl*: plates from EtOH. M.p. 165-6°.  
Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. boiling EtOH.

N'-Naphthyl: *see sym.*-Di-2-naphthylthiourea.N'-*Acetyl*: prisms from EtOH. M.p. 158°.

See first reference above and also

Cosiner, *Ber.*, 1881, 14, 61. $\alpha$ -Naphthylurea $C_{11}H_{10}ON_2$ 

MW, 186

Needles from EtOH. M.p. 215-20°. Very  
sol. EtOH. Sol.  $Et_2O$ . Spar. sol.  $H_2O$ . Above  
m.p.  $\rightarrow$  *sym.*-di-1-naphthylurea.

N'-*Phenyl*: m.p. 222-3°.N'-*p-Tolyl*: needles. M.p. 234°.N'-*Benzyl*: leaflets from EtOH. M.p. 203°.

N'-*Acetyl*: needles. M.p. 214-15°. Mod. sol.  
EtOH,  $C_6H_6$ .

N'-*Benzoyl*: exists in two forms. (i) Needles.  
M.p. 243-243.5°. (ii) Prisms. M.p. 165-6°.

N'-*Carboethoxyl*: needles from EtOH or  $C_6H_6$ .  
M.p. 170-170.5°.

N'- $\alpha$ -Naphthyl: *see sym.*-Di-1-naphthylurea.Young, Clark, *J. Chem. Soc.*, 1897, 71, 1200.Walther, Wlodkowski, *J. prakt. Chem.*, 1899, 59, 277.Sah, *Chem. Abstracts*, 1934, 28, 6122.

**$\beta$ -Naphthylurea.**

Needles from EtOH. M.p. 219–20° (213–14°). Sol. hot EtOH. Insol. cold H<sub>2</sub>O. Above m.p.  $\rightarrow$  *sym.*-di-2-naphthylurea.

N'-*Phenyl*: prisms from EtOH. M.p. 220–1°.

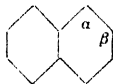
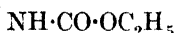
N'-*Acetyl*: needles. M.p. 202–202.5°. Sol. boiling EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. boiling H<sub>2</sub>O.

N'-*Benzoyl*: needles from EtOH. M.p. 219–20°.

N'-*Carboethoxyl*: needles from EtOH. M.p. 140°.

N'-*Naphthyl*: see Di-2-naphthylurea.

See previous references.

 **$\alpha$ -Naphthylurethane**

C<sub>13</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 215

Needles from EtOH. M.p. 79°.

Hofmann, *Ber.*, 1870, 3, 657.

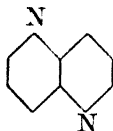
 **$\beta$ -Naphthylurethane.**

Needles. M.p. 73°. Sol. boiling H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.

Cosiner, *Ber.*, 1881, 14, 60.

Groeneveld, *Rec. trav. chim.*, 1932, 51, 783.

**1 : 5-Naphthyridine** (1 : 5-Pyridopyridine, *isonaphthyridine*)



C<sub>8</sub>H<sub>6</sub>N<sub>2</sub>

MW, 130

Yellowish needles from pet. ether. M.p. 75°. B.p. 112°/15 mm. Very sol. all solvents. Aq. sol. has bitter taste and reacts neutral. Sublimes very readily. Volatile in steam.

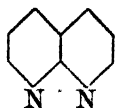
B, H<sub>2</sub>SO<sub>4</sub>: m.p. about 218° decomp. Sol. H<sub>2</sub>O, EtOH.

*Picrate*: m.p. 200°. Sol. H<sub>2</sub>O, EtOH.

Schering-Kahlbaum, D.R.P., 507,637, (*Chem. Abstracts*, 1931, 25, 716).

Bobrański, Sucharda, *Ber.*, 1927, 60, 1081.

**1 : 8-Naphthyridine** (1 : 8-Pyridopyridine)



C<sub>8</sub>H<sub>6</sub>N<sub>2</sub>

MW, 130

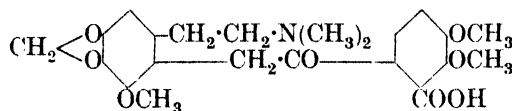
Needles. M.p. 98–9°. Sublimes at 80°/13 mm. Very hygroscopic. Bitter taste. Reacts with dil. KMnO<sub>4</sub>.

*Picrate*: yellow needles from EtOH. M.p. 207–8°.

*Methiodide*: orange-yellow needles from EtOH. M.p. 180–1°. Sol. H<sub>2</sub>O. Less sol. EtOH.

Koller, *Ber.*, 1927, 60, 1918.

**Narceine** (3 : 4 : 6'-Trimethoxy-4' : 5'-methylenedioxy-2'-[ $\beta$ -dimethylaminoethyl]deoxybenzoic-2-carboxylic acid)



C<sub>23</sub>H<sub>27</sub>O<sub>8</sub>N

MW, 445

Needles or prisms + 3H<sub>2</sub>O from boiling H<sub>2</sub>O. M.p. 171° (176–7°), anhyd. 145–2°. Sol. 769 parts H<sub>2</sub>O at 25°, 1285 parts H<sub>2</sub>O at 13°, 945 parts 80% EtOH at 13°. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, pet. ether. Sol. NH<sub>3</sub>.Aq., dil. alkalis. Weak base. NaOH + Me<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  narceonic acid. Dil. iodine sol. gives blue col. Dil. H<sub>2</sub>SO<sub>4</sub> + resorcinol  $\rightarrow$  red col. on heating. Tannin + H<sub>2</sub>SO<sub>4</sub> on heating  $\rightarrow$  green col.  $\rightarrow$  yellowish-brown on cooling.

B, HCl: m.p. 192°.

B, H<sub>2</sub>AuCl<sub>4</sub>: yellow needles from EtOH. M.p. 130°.

B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>: golden-yellow needles. M.p. 195–6°.

B, HgCl<sub>2</sub>: m.p. 120–3°.

*Picrate*: m.p. 195°.

*Methochloride*: cryst. from EtOH. M.p. 243°. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

*Methiodide*: needles. M.p. 207°.

*Methochloroplatinate*: yellow leaflets from EtOH.Aq. M.p. 209–10°.

*Methylbetaine*: m.p. 266° decomp. Very sol. H<sub>2</sub>O. Spar. sol. EtOH, Et<sub>2</sub>O.

*Ethochloride*: m.p. 231°. Very sol. H<sub>2</sub>O. Sol. EtOH. Insol. Et<sub>2</sub>O.

*Ethochloroplatinate*: m.p. 181–2°.

*Ethylbetaine*: m.p. 175–7°.

*Me ester*: C<sub>24</sub>H<sub>29</sub>O<sub>8</sub>N. MW, 459. B, HCl: plates from H<sub>2</sub>O. M.p. 151–2°. B, HBr: prisms from H<sub>2</sub>O. M.p. 153–4°. B, HI: m.p. 181–2°. B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>: m.p. 205–6°. *Methiodide*: needles. M.p. 211°. *Methosulphate*: m.p. 213–14°.

*Et ester*: C<sub>25</sub>H<sub>31</sub>O<sub>8</sub>N. MW, 473. B, HCl: prisms. M.p. 208–10°. Sol. EtOH, CHCl<sub>3</sub>. Sol. 120 parts cold H<sub>2</sub>O. B, HBr: prisms from

H<sub>2</sub>O. M.p. 215–16°. *B,HI*: prisms. M.p. 212–13°. *B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 194–5°. *Methochloride*: m.p. 214–16°. Sol. H<sub>2</sub>O, EtOH. *Methiodide*: m.p. 209–10°. *Methochloroplatinate*: m.p. 220°. *Ethochloride*: m.p. 218–19°. Sol. H<sub>2</sub>O. *Ethiodide*: cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 141°. *Ethochloroplatinate*: m.p. 220°.

*Isopropyl ester*: *B,HI*, m.p. 224–5°.

*Butyl ester*: *B,HI*, m.p. 185–6°.

*Diethylaminoethyl ester*: C<sub>29</sub>H<sub>40</sub>O<sub>8</sub>N<sub>2</sub>. MW, 544. Prisms from MeOH. M.p. 203°.

*Amide*: C<sub>23</sub>H<sub>28</sub>O<sub>7</sub>N<sub>2</sub>. MW, 444. Cryst. + H<sub>2</sub>O from EtOH.Aq. M.p. 178°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O. *B,HCl*: plates. M.p. 236–7°. Sol. hot H<sub>2</sub>O. *B,HI*: prisms. M.p. 216–18°.

*Oxime*: prisms + H<sub>2</sub>O from 80% EtOH. Decomp. at 167°. Very sol. alkalis. *Me ether*: cryst. from MeOH. M.p. 190°.

Addinall, Major, *J. Am. Chem. Soc.*, 1933, **55**, 1202, 2153.

Freund, Frankforter, *Ann.*, 1893, **277**, 25.

Freund, *Ber.*, 1907, **40**, 194.

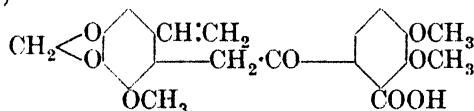
Freund, Michaelis, *Ann.*, 1895, **286**, 250.

Tambach, Jäger, *Ann.*, 1906, **349**, 191.

### Narceol.

See under *p*-Cresol.

**Narceonic Acid** (3 : 4 : 6'-Trimethoxy-4' : 5'-methylenedioxy-2'-vinyldeoxybenzoin-2-carboxylic acid)



C<sub>21</sub>H<sub>20</sub>O<sub>8</sub> MW, 400

Plates from EtOH-CHCl<sub>3</sub>. M.p. 217°. Sol. CHCl<sub>3</sub>. Less sol. EtOH, AcOH. Insol. H<sub>2</sub>O.

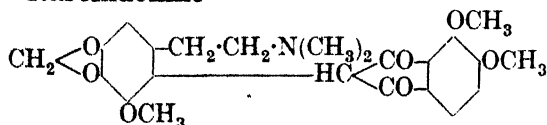
*Me ester*: C<sub>22</sub>H<sub>22</sub>O<sub>8</sub>. MW, 414. Cryst. from MeOH. M.p. 155°.

*Et ester*: C<sub>23</sub>H<sub>24</sub>O<sub>8</sub>. MW, 428. Cryst. from EtOH. M.p. 139–40°.

Addinall, Major, *J. Am. Chem. Soc.*, 1933, **55**, 1207.

Freund, *Ann.*, 1893, **277**, 56.

### Narcindonine



C<sub>23</sub>H<sub>25</sub>O<sub>7</sub>N MW, 427

Red cryst. + 1½ H<sub>2</sub>O from H<sub>2</sub>O. M.p. 174°. Sol. hot H<sub>2</sub>O, EtOH, MeOH, warm CHCl<sub>3</sub>, AcOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

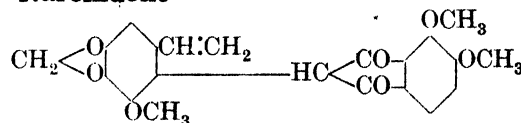
*B,HCl*: cryst. M.p. 255°. Mod. sol. H<sub>2</sub>O. Spar. sol. EtOH.

*B,HI*: cryst. M.p. 246°.

*Methiodide*: leaflets from EtOH. M.p. 217°. Mod. sol. EtOH, AcOH. Less sol. H<sub>2</sub>O. Sol. alkalis with red col. Warm saturated NaOEt sol. → narcindone. *Me ether*: yellowish-red platelets from EtOH. M.p. 207°. Sol. MeOH, EtOH. Mod. sol. H<sub>2</sub>O. Insol. Et<sub>2</sub>O.

Freund, Oppenheim, *Ber.*, 1909, **42**, 1092.

### Narcindone



C<sub>21</sub>H<sub>18</sub>O<sub>7</sub> MW, 382

Leaflets from AcOH. M.p. 136–7°. Very sol. MeOH, EtOH, AcOH. Sol. alkalis with red col. Insol. NH<sub>3</sub>.Aq., Na<sub>2</sub>CO<sub>3</sub>.

Freund, Oppenheim, *Ber.*, 1909, **42**, 1094.

### Narcipoetine

C<sub>18</sub>H<sub>22</sub>O<sub>4</sub>N MW, 316

Alkaloid from *Narcissus poeticus*, Linn. Silky needles from EtOH.Aq. M.p. 172°. [ $\alpha$ ]<sub>D</sub> + 84.4° in 96% EtOH.

*B,HCl*: cryst. + H<sub>2</sub>O. M.p. 271° decomp. [ $\alpha$ ]<sub>D</sub> + 111.2° in 96% EtOH.

*B,HAuCl<sub>4</sub>*: yellow cryst. M.p. 131–2° decomp.

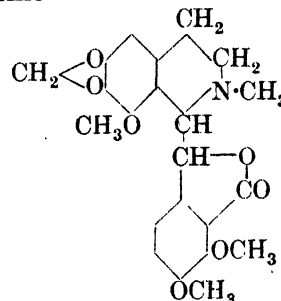
*Picrate*: yellow cryst. from EtOH. M.p. 261° decomp.

Kolle, Gloppe, *Chem. Zentr.*, 1934, **II**, 261.

### Narcissine.

See Lycorine.

### $\alpha$ -Narcotine



C<sub>22</sub>H<sub>23</sub>O<sub>7</sub>N MW, 413

*l*,

One of the opium alkaloids. Needles from hot EtOH. M.p. 176°. Sol. CHCl<sub>3</sub>. Sol. 166 parts

Et<sub>2</sub>O, 100 parts 80% EtOH, 24 parts Me<sub>2</sub>CO, 31 parts AcOEt, 22 parts C<sub>6</sub>H<sub>6</sub>, 25 parts aniline, 2·3 parts Py, 1·7 parts piperidine, 0·4 parts diethylamine, at 16° C. Insol. H<sub>2</sub>O. alkalis.  $[\alpha]_D -198\cdot0^\circ$  in CHCl<sub>3</sub>, + 50° in 1% HCl. Salts easily decomp. by H<sub>2</sub>O. Heat of comb. C<sub>p</sub> 2645 Cal.  $k = 1\cdot5 \times 10^{-8}$  at 25°. Conc. H<sub>2</sub>SO<sub>4</sub> → pale yellow → yellowish-red col. HNO<sub>3</sub> → yellowish-red col. → colourless on standing. Dil. H<sub>2</sub>SO<sub>4</sub> or Ba(OH)<sub>2</sub> → hydrocotarnine + opianic acid. HNO<sub>3</sub> → opianic acid + cotarnine.

N-Oxide : solid.  $[\alpha]_D +135^\circ$  in CHCl<sub>3</sub>. Very sol. H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Hygroscopic. Unstable to heat. SO<sub>2</sub> → narcotine. Gives red col. with acetic anhydride. B.HCl : m.p. 193°. Very sol. H<sub>2</sub>O.  $[\alpha]_D +100^\circ$ . Picrate : m.p. 130°. Chloroplatinate : m.p. 175°.

Picrate : m.p. 174°.

d-Bromocamphorsulphonate : needles from EtOH or AcOEt. M.p. 110–20°.  $[\alpha]_D +100\cdot7^\circ$  in CHCl<sub>3</sub>.

l-Bromocamphorsulphonate : prisms from AcOEt. M.p. 180–5°.  $[\alpha]_D +29^\circ$  in CHCl<sub>3</sub>.

dl.

Needles from EtOH. M.p. 175°.  $[\alpha]_D +199\cdot9^\circ$  in CHCl<sub>3</sub>.

d-Bromocamphorsulphonate : prisms from AcOEt. M.p. 170–85°.

l-Bromocamphorsulphonate : m.p. 80–90°.  $[\alpha]_D -97\cdot2^\circ$  in CHCl<sub>3</sub>.

dl. α-Gnoscopine.

Needles from EtOH. M.p. 232–3°. Sol. hot CHCl<sub>3</sub>. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Very spar. sol. EtOH. Much less sol. than active forms.

Picrate : yellow prisms from MeOH. M.p. 188–9°.

Methiodide : prisms + 2H<sub>2</sub>O. M.p. 210–12°.

dl-Bromocamphorsulphonate : prisms from AcOEt. M.p. 189°.

Polonovski, Polonovski, *Bull. soc. chim.*, 1930, 47, 361.

Marshall, Pyman, Robinson, *J. Chem. Soc.*, 1934, 1317.

Perkin, Robinson, *J. Chem. Soc.*, 1911, 99, 775.

Hesse, *Ann.*, 1872, 8, 284 (Suppl.).

### β-Narcotine.

Stereoisomer of α-narcotine.

dl.

Plates from EtOH. M.p. 176°.  $[\alpha]_{546}^{18} +103^\circ$  in CHCl<sub>3</sub>.

l.

Leaflets or plates from EtOH, prisms from

Me<sub>2</sub>CO. M.p. 176°.  $[\alpha]_{546}^{18} -101^\circ$  in CHCl<sub>3</sub>, – 60° in 1% HCl. Much less readily racemised than α-form.

Picrate : yellow needles from EtOH. M.p. 118°.

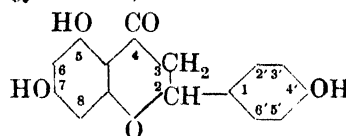
Methiodide : prisms from hot H<sub>2</sub>O. M.p. 208°.

dl.

See β-Gnoscopine.

See second reference above.

**Naringenin** (*Naringetol*, *salipuro*l, 5 : 7 : 4'-*Trihydroxyflavanone*)



C<sub>15</sub>H<sub>12</sub>O<sub>5</sub>

MW, 272

Needles or leaflets from EtOH.Aq. M.p. 251°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. Alkalis → yellow sols. which are decomp. by CO<sub>2</sub>. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. changing to red on standing. Alc. FeCl<sub>3</sub> → deep reddish-brown col. Mg + alc. HCl → reddish-violet col. Heat with conc. KOH → phloroglucinol + *p*-coumaric acid.

4'-Me ether : see Isosakuranetin.

7-Me ether : see Sakuranetin.

7 : 4'-Di-Me ether : isosakuranetin Me ether. See under Isosakuranetin.

Tri-Me ether : isosakuranetin di-Me ether. See under Isosakuranetin.

5-Rhamnoglucoside : see Naringin.

Oxime : needles from EtOH.Aq. M.p. 233°.

Triacetyl : cryst. from AcOH. M.p. 53–5°. No col. with FeCl<sub>3</sub>. Mg + alc. HCl → deep red col. Acetic anhydride + Na acetate → 4 : 2' : 4' : 6'-tetracetoxychalkone.

Tetra-acetyl : cryst. from EtOH. M.p. 133–6°.

Shibata, Nagai, *Acta Phytotechim.*, 1924, 2, 37.

Shinoda, Uyeda, *Chem. Zentr.*, 1929, II, 1547.

Asahina, Shinoda, Inubuse, *Chem. Zentr.*, 1928, II, 49.

Asahina, Inubuse, *Ber.*, 1928, 61, 1514.

Rosenmund, Rosenmund, *ibid.*, 2608.

**Naringin** (*Naringenin-5-rhamnoglucoside*)

C<sub>27</sub>H<sub>32</sub>O<sub>14</sub>

MW, 580

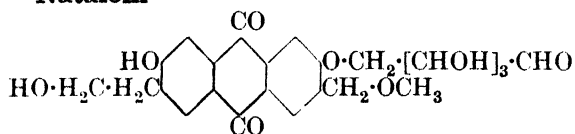
Found in fruits of *Citrus decumana*. Needles + 8H<sub>2</sub>O from H<sub>2</sub>O. M.p. 82°. After drying at 110° contains 2H<sub>2</sub>O and has m.p. 171°. Very sol.

hot EtOH. Sol. hot H<sub>2</sub>O, AcOH. Sol. 8000 parts H<sub>2</sub>O at 20°. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.  $[\alpha]_D^{20} - 82.11^\circ$  in EtOH. Bitter taste. Gives intense yellowish-red col. with alkalis. Alc. FeCl<sub>3</sub>  $\rightarrow$  brownish-red col. Hyd.  $\rightarrow$  naringenin + glucose + rhamnose.

Asahina, Inubuse, *Chem. Zentr.*, 1929, I, 2429; *Ber.*, 1928, **61**, 1514.

Zoller, *Chem. Zentr.*, 1918, II, 635.

### Nataloin



C<sub>23</sub>H<sub>24</sub>O<sub>10</sub>

MW, 460

*l.*

Pale yellow plates from MeOH.  $[\alpha]_D^{18} - 145^\circ$ . Sol. AcOEt, Py. Mod. sol. MeOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O. Sol. HCl, HNO<sub>3</sub>. Alk. sols. decomp. by CO<sub>2</sub>. Conc. H<sub>2</sub>SO<sub>4</sub> + fuming HNO<sub>3</sub>  $\rightarrow$  green col. NaOH + ammonium persulphate  $\rightarrow$  violet col.

$\alpha$ -Penta-acetyl: m.p. 198°.  $[\alpha]_D^{20} - 53^\circ$  in EtOH,  $- 50^\circ$  in AcOH. Hyd.  $\rightarrow$  *l*-nataloin.

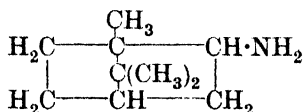
$\beta$ -Penta-acetyl:  $[\alpha]_D - 44^\circ$ . Hyd.  $\rightarrow$  *l*-nataloin.

*dl.*

Penta-acetyl: m.p. 245° decomp. Hyd.  $\rightarrow$  *l*-nataloin + mixture of *d*- and *l*-nataloin,  $[\alpha]_D + 63^\circ$ .

Léger, *J. pharm. chim.*, 1903, **17**, 13.

### Neobornylamine



C<sub>10</sub>H<sub>19</sub>N

MW, 153

*d.*

Powder. M.p. 184°. Very sol. all org. solvents. Insol. H<sub>2</sub>O.  $[\alpha]_D - 43.7^\circ$  in EtOH,  $- 27^\circ$  in C<sub>6</sub>H<sub>6</sub>.

*B*, HCl: needles from H<sub>2</sub>O. Does not melt below 320°. Very sol. H<sub>2</sub>O, EtOH. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>.  $[\alpha]_D - 44.2^\circ$  in EtOH.

*B*<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>: orange leaflets from EtOH. M.p. 303° decomp. Very sol. hot EtOH. Sol. hot H<sub>2</sub>O.

*Picrate*: yellow prisms from EtOH. M.p. 248° decomp. Sol. hot EtOH.

*Formyl*: leaflets from EtOH.Aq. M.p. 72–3°.  $[\alpha]_D^{20} - 19.4^\circ$  in EtOH. Very sol. EtOH, pet. ether.

*Acetyl*: needles from pet. ether. M.p. 144°.  $[\alpha]_D - 19.5^\circ$  in EtOH. Sol. EtOH. Spar. sol. pet. ether.

*Benzoyl*: leaflets from EtOH.Aq. M.p. 130°. Sol. EtOH. Spar. sol. cold pet. ether.

*Urethane*: cryst. M.p. 36°.  $[\alpha]_D - 9.63^\circ$  in C<sub>6</sub>H<sub>6</sub>. Sol. org. solvents. Spar. sol. H<sub>2</sub>O.

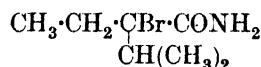
Forster, *J. Chem. Soc.*, 1898, **73**, 394.

Forster, Hart-Smith, *J. Chem. Soc.*, 1900, **77**, 1152.

### Neocinchophene.

See under 6-Methyl-2-phenylquinoline-4-carboxylic Acid.

**Neodorme** (1-Bromo-1-isopropylbutyramide)



C<sub>7</sub>H<sub>14</sub>ONBr

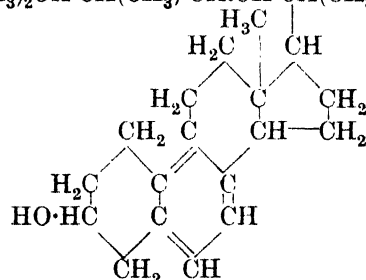
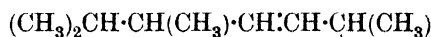
MW, 208

Cryst. M.p. 50–1°. Odour resembling menthol. Sol. 150 parts cold H<sub>2</sub>O. Readily sol. most org. solvents. Sublimes in needles. Aq. sols. react acid. Decomp. by boiling H<sub>2</sub>O and hot aq. alkalis. Hypnotic and sedative.

Kuhlmann, *Pharm. Ztg.*, 1931, **76**, 113.

Knoll, U.S.P., 1,780,131, (*Chem. Abstracts*, 1931, **25**, 1038).

### Neoergosterol



C<sub>27</sub>H<sub>42</sub>O

MW, 382

Prisms from EtOH. M.p. 151–2°.  $[\alpha]_D - 12^\circ$  in CHCl<sub>3</sub>. Does not give Salkowski or Liebermann-Burchard colour reactions.

*Acetyl*: needles from Me<sub>2</sub>CO. M.p. 122–3°. *Dibromide*: cryst. from MeOH–Me<sub>2</sub>CO. M.p. 183°.

3 : 5-Dinitrobenzoyl: cryst. from C<sub>6</sub>H<sub>6</sub>–EtOH. M.p. 218–20°.  $[\alpha]_D^{18} - 13^\circ$  in CHCl<sub>3</sub>.

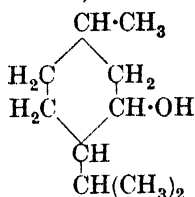
*Dibromide*: m.p. 212° decomp.

Windaus, Borgeaud, *Ann.*, 1928, **460**, 235.

Bonstedt, *Z. physiol. Chem.*, 1929, **185**, 165.

Inhoffen, *Ann.*, 1932, **497**, 130.

**Neoisomenthol** (3-Methyl-6-isopropylcyclohexanol, p-menthanol-3)



$C_{10}H_{20}O$

MW, 156

*d*-.  
F.p.  $-8^{\circ}$ . B.p.  $214.6^{\circ}/760$  mm.,  $84.2^{\circ}/7.5$  mm.  $D_4^{20}$  0.9131.  $[\alpha]_D^{25} + 2.2^{\circ}$  in EtOH.  $n_D^{18}$  1.4674. Easily oxidised to *d*-isomenthone.

*p*-Nitrobenzoyl: pale yellow prisms from EtOH. M.p.  $72.5-73^{\circ}$ .  $[\alpha]_D^{25} - 5.3^{\circ}$  in  $CHCl_3$ .

3:5-Dinitrobenzoyl: needles from EtOH-AcOEt. M.p.  $100.5-101^{\circ}$ .  $[\alpha]_D^{25} - 9.5^{\circ}$  in  $CHCl_3$ .

*d*-Camphor-10-sulphonate: prisms from pet. ether. M.p.  $69-70^{\circ}$ .  $[\alpha]_D^{25} + 17.3^{\circ}$  in  $CHCl_3$ .

1-Camphor-10-sulphonate: needles from pet. ether. M.p.  $84-6^{\circ}$ .  $[\alpha]_D^{25} - 41.0^{\circ}$  in  $CHCl_3$ .

$B, H_3PO_4$ : m.p.  $60^{\circ}$ .

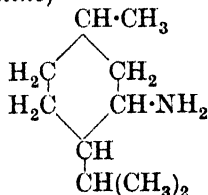
*dl*-.  
F.p.  $14^{\circ}$ . B.p.  $81^{\circ}/6$  mm.  $n_D^{17}$  1.4676.

*p*-Nitrobenzoyl: yellow needles from EtOH. M.p.  $63-4^{\circ}$ .

3:5-Dinitrobenzoyl: leaflets from EtOH. M.p.  $73-73.5^{\circ}$ .

Read, Grubb, *J. Chem. Soc.*, 1934, 315.

**Neoisomenthylamine** (3-Methyl-6-isopropylcyclohexylamine)



$C_{10}H_{21}N$

MW, 155

*d*-.  
Liq.  $[\alpha]_D^{25} + 9^{\circ}$  in  $CHCl_3$ .

$B, HCl$ : needles from  $Me_2CO-MeOH$ . Does not melt below  $250^{\circ}$ .  $[\alpha]_D^{25} + 20.9^{\circ}$  in  $H_2O$ .

Formyl: oil.  $[\alpha]_D - 3.9^{\circ}$  in  $CHCl_3$ .

Acetyl: needles from pet. ether. M.p.  $99-100^{\circ}$ .  $[\alpha]_D^{25} - 2.6^{\circ}$  in  $CHCl_3$ . Very sol. org. solvents.

Benzoyl: needles from EtOH. M.p.  $151^{\circ}$ .  $[\alpha]_D^{25} - 10.4^{\circ}$  in  $CHCl_3$ .

2-Naphthalenesulphonyl: cryst. from EtOH. M.p.  $120^{\circ}$ .  $[\alpha]_D^{25} - 10.7^{\circ}$  in  $CHCl_3$ .

Benzylidene: needles from MeOH. M.p.  $68-9^{\circ}$ .  $[\alpha]_D^{25} - 34.2^{\circ}$  in  $CHCl_3$ .

Salicylidene: yellow needles from  $Et_2O$ -pet. ether. M.p.  $99-100^{\circ}$ .  $[\alpha]_D^{25} - 17.9^{\circ}$  in  $CHCl_3$ .

Carbamide: needles from  $Et_2O$ -pet. ether. M.p.  $115-16^{\circ}$ .  $[\alpha]_D^{25} - 3.1^{\circ}$  in  $CHCl_3$ .

Phenylcarbamide: needles from  $Et_2O$ -pet. ether. M.p.  $149-50^{\circ}$ .  $[\alpha]_D^{25} - 12.1^{\circ}$  in  $CHCl_3$ .

Phenylthiocarbamide: prisms from  $Et_2O$ -pet. ether. M.p.  $99^{\circ}$ .  $[\alpha]_D^{25} - 6.7^{\circ}$  in  $CHCl_3$ .

Read, Robertson, *J. Chem. Soc.*, 1927, 2168.

### Neolactose

$C_{12}H_{22}O_{11}$

MW, 342

Constitution unknown. Prisms from MeOH. M.p.  $190^{\circ}$  decomp.  $[\alpha]_D^{20} + 34.6^{\circ} \rightarrow + 35.5^{\circ}$  in  $H_2O$ . Hyd.  $\rightarrow d$ -galactose + *d*-altrose. Br water  $\rightarrow$  neolactobionic acid.  $HNO_3 \rightarrow$  mucic acid.

Phenylosazone: yellow cryst. from  $H_2O$ . M.p.  $195^{\circ}$  decomp.

$\beta$ -Hepta-acetyl: prisms. M.p.  $135-6^{\circ}$ .  $[\alpha]_D + 10.0^{\circ} \rightarrow + 21.0^{\circ}$  in  $CHCl_3$ .

$\alpha$ -Octa-acetyl: laminae from EtOH. M.p.  $178^{\circ}$ .  $[\alpha]_D^{24} + 53.4^{\circ}$  in  $CHCl_3$ . Reduces hot Fehling's.

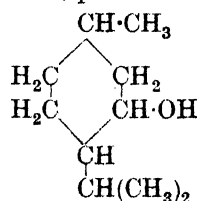
$\beta$ -Octa-acetyl: plates from EtOH. M.p.  $148^{\circ}$ .  $[\alpha]_D^{23} - 7.04^{\circ}$  in  $CHCl_3$ .

$\alpha$ -Chlorohepta-acetyl: m.p.  $182^{\circ}$  decomp.  $[\alpha]_D^{25} + 71.2^{\circ}$  in  $CHCl_3$ .

Kunz, Hudson, *J. Am. Chem. Soc.*, 1926, 48, 1978, 2435.

Richtmyer, Hudson, *J. Am. Chem. Soc.*, 1935, 57, 1716.

**Neomenthol** ( $\beta$ -Pulegomenthol, 3-methyl-6-isopropylcyclohexanol, p-menthanol-3)



$C_{10}H_{20}O$

MW, 156

*d*-.  
Found in Japanese peppermint oil. Liq. F.p.  $-22^{\circ}$ . B.p.  $87^{\circ}/8$  mm.  $n_D^{17}$  1.4617.  $[\alpha]_D^{16} + 17.80^{\circ}$  in EtOH.

Benzoyl: cryst. from EtOH. M.p.  $68-9^{\circ}$ . B.p.  $181^{\circ}/16$  mm.  $[\alpha]_{D_{44}} + 32.3^{\circ}$  in  $CHCl_3$ .

*p*-Nitrobenzoyl: cryst. from EtOH.Aq. M.p.  $94.5-95^{\circ}$ .  $[\alpha]_D^{25} + 17.8^{\circ}$  in  $CHCl_3$ .

1-Menthoxycetate: m.p.  $28.5^{\circ}$ .  $[\alpha]_D^{15} - 31.5^{\circ}$  in  $CHCl_3$ .

*d*-Menthoxycetate: m.p.  $64^{\circ}$ .  $[\alpha]_D^{16} + 80.9^{\circ}$  in  $CHCl_3$ .

*Acid phthalate*: cryst. from AcOH. M.p. 142-4°.  $[\alpha]_D^{20} + 68.7^\circ$  in  $\text{CHCl}_3$ . *Brucine salt*: cryst. from  $\text{Me}_2\text{CO}$ . M.p. 125-7° decomp.

*l.*

Liq. B.p. 97.6°/10 mm.  $n_D^{15} 1.4638$ .  $[\alpha]_D^{17} - 20.7^\circ$  in EtOH.

*p-Nitrobenzoyl*: yellow needles from EtOH. Aq. M.p. 95°.  $[\alpha]_D^{18} - 17.9^\circ$  in  $\text{CHCl}_3$ .

*3:5-Dinitrobenzoyl*: yellow needles from AcOEt-EtOH. M.p. 153°.  $[\alpha]_D^{18} - 23.9^\circ$  in  $\text{CHCl}_3$ .

*Phenylurethane*: m.p. 107-8°.  $[\alpha]_D - 26.77^\circ$  in  $\text{CHCl}_3$ .

*l-Menthoxycetate*: needles from MeOH. M.p. 64°.  $[\alpha]_D^{18} - 81.1^\circ$  in  $\text{CHCl}_3$ .

*d-Menthoxycetate*: needles. M.p. 28.5°.  $[\alpha]_D^{19} + 32.0^\circ$  in  $\text{CHCl}_3$ .

*d-Camphor-10-sulphonate*: long needles from pet. ether. M.p. 116° decomp.  $[\alpha]_D^{18} + 8.9^\circ$  in  $\text{CHCl}_3$ .

*l-Camphor-10-sulphonate*: cryst. from pet. ether. M.p. 92° decomp.  $[\alpha]_D^{18} - 50.3^\circ$  in  $\text{CHCl}_3$ .

*dl.*

Plates from pet. ether. M.p. 51°. B.p. 212.1°/163 mm., 103.5°/16 mm. Sol. org. solvents. Insol.  $\text{H}_2\text{O}$ .  $\text{CrO}_3 \rightarrow$  *dl*-menthone.

*p-Nitrobenzoyl*: needles from warm EtOH. M.p. 78.5°.

*3:5-Dinitrobenzoyl*: yellow needles from EtOH-AcOEt. M.p. 130°. Mod. sol. hot EtOH.

*2-Naphthoyl*: prisms from EtOH. M.p. 98°.

*Phenylurethane*: m.p. 114°.

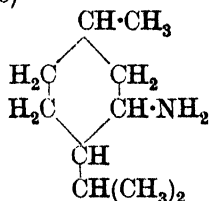
*Acid phthalate*: prisms from AcOH. M.p. 175-7°.

Pickard, Littlebury, *J. Chem. Soc.*, 1912, 101, 110.

Read, Grubb, *J. Chem. Soc.*, 1933, 167.

Read, Grubb, *J. Soc. Chem. Ind.*, 1934, 53, 52T.

**Neomenthylamine** (3-Methyl-6-isopropyl-cyclohexylamine)



$\text{C}_{10}\text{H}_{21}\text{N}$

*d.*

Liq.  $[\alpha]_D^{20} + 15.73^\circ$  in  $\text{CHCl}_3$ .

*B,HCl*: prisms from pet. ether.. M.p. 189°.  $[\alpha]_D^{18} + 21.5^\circ$  in  $\text{H}_2\text{O}$ .

*Formyl*: cryst. from MeOH. M.p. 117-18°.

MW, 155

$[\alpha]_D^{18} + 53.8^\circ$  in  $\text{CHCl}_3$ ,  $+ 62.4^\circ$  in EtOH. Mod. sol.  $\text{Et}_2\text{O}$ , pet. ether.

*Acetyl*: m.p. 169-70°.  $[\alpha]_D^{18} + 53.0^\circ$  in  $\text{CHCl}_3$ .

*Benzoyl*: m.p. 121.5°.  $[\alpha]_D^{18} + 22.7^\circ$  in  $\text{CHCl}_3$ .

*2-Naphthalenesulphonyl*: leaflets from EtOH- $\text{CHCl}_3$ . M.p. 208°.  $[\alpha]_D^{18} + 43.7^\circ$  in  $\text{CHCl}_3$ .

*Benzylidene*: m.p. 45-6°.  $[\alpha]_D^{18} + 61.7^\circ$  in  $\text{CHCl}_3$ .

*Salicylidene*: m.p. 99-100°.  $[\alpha]_D^{18} + 30.0^\circ$  in  $\text{CHCl}_3$ .

*l.*

*Formyl*: cryst. from EtOH. M.p. 116-17°  $[\alpha]_D^{18} - 53.6^\circ$  in  $\text{CHCl}_3$ .

*dl.*

*B,HCl*: cryst. from pet. ether. M.p. 184-5°. Sol. most org. solvents.

*B,HBr*: prisms from EtOH. Does not melt below 220°. Spar. sol.  $\text{Et}_2\text{O}$ . Insol. pet. ether.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange prisms from  $\text{H}_2\text{O}$ . M.p. 206-7° decomp.

*Picrate*: yellow needles or orange prisms from EtOH. M.p. 183-4°.

*Formyl*: prisms from  $\text{Et}_2\text{O}$ -pet. ether. M.p. 86°. Sol. most org. solvents. More sol. than *d*-form.

*Acetyl*: needles from  $\text{Et}_2\text{O}$ -pet. ether. M.p. 160-1°.

*Benzoyl*: needles from pet. ether. M.p. 101-2°.

*2-Naphthalenesulphonyl*: plates from boiling MeOH. M.p. 209-10°. Spar. sol. MeOH.

*Salicylidene*: yellow needles from MeOH. M.p. 69.5°.

*Carbamide*: prisms from  $\text{Me}_2\text{CO}$ . M.p. 162-3°.

*Phenylcarbamide*: prisms from MeOH. M.p. 183-4°.

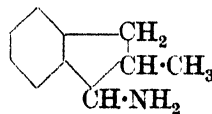
*Phenylthiocarbamide*: prisms from MeOH. M.p. 169-70°.

Read, Robertson, *J. Chem. Soc.*, 1926, 2218.

Read, Cook, Shannon, *ibid.*, 2227.

See also Tutin, Kipping, *J. Chem. Soc.*, 1904, 85, 68.

**Neo-2-methyl-1-hydrindamine** (*Neo-2-methyl-1-indanamine*)



$\text{C}_{10}\text{H}_{13}\text{N}$

*d.*

*B,HCl*:  $[\alpha]_D + 3.1^\circ$  in  $\text{H}_2\text{O}$ .

*Acid-d-tartrate*: prisms +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .

MW, 147



M.p. 166–7°. Very sol.  $\text{H}_2\text{O}$ .  $[\alpha]_D + 16.2^\circ$  in  $\text{H}_2\text{O}$ .

d-Camphorsulphonate: needles from  $\text{H}_2\text{O}$ . Decomp. at 195–205°.  $[\alpha]_D + 14.8^\circ$ .

3-Bromo-d-camphor-8-sulphonate: needles +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. 229–30°. Sol. EtOH. Mod. sol.  $\text{H}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ , AcOEt. Insol.  $\text{Et}_2\text{O}$ ,  $\text{CCl}_4$ .

l.

B.HCl: needles from  $\text{H}_2\text{O}$ . Decomp. at 235°. Very sol.  $\text{H}_2\text{O}$ , EtOH. Sol.  $\text{CHCl}_3$ , AcOEt. Almost insol.  $\text{Et}_2\text{O}$ ,  $\text{CCl}_4$ .  $[\alpha]_D - 3.1^\circ$  in  $\text{H}_2\text{O}$ .

Acid-d-tartrate: prisms +  $\text{H}_2\text{O}$  from EtOH or  $\text{H}_2\text{O}$ . M.p. 173°. Sol.  $\text{H}_2\text{O}$ . Less sol. EtOH. Almost insol.  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , AcOEt.  $[\alpha]_D + 11.4^\circ$  in  $\text{H}_2\text{O}$ .

d-Camphorsulphonate: prisms from  $\text{H}_2\text{O}$ . M.p. 220°. Very sol.  $\text{H}_2\text{O}$ . Sol.  $\text{CHCl}_3$ . Spar. sol. EtOH,  $\text{Me}_2\text{CO}$ . Insol.  $\text{Et}_2\text{O}$ , AcOEt.  $[\alpha]_D + 11.6^\circ$  in  $\text{H}_2\text{O}$ .

3-Bromo-d-camphor-8-sulphonate: needles from  $\text{H}_2\text{O}$ . M.p. 214°. Sol. EtOH, AcOEt,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Mod. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ .  $[\alpha]_D + 57.1^\circ$  in  $\text{H}_2\text{O}$ .

Benzoyl: needles from EtOH.Aq. M.p. 171°.

dl.

B.HCl: needles from  $\text{H}_2\text{O}$ . Decomp. at 235°.

$\text{B}_2\text{H}_2\text{SO}_4$ : plates from  $\text{H}_2\text{O}$ . M.p. about 220° decomp.

Acid oxalate: prisms +  $2\text{H}_2\text{O}$  from EtOH.Aq. M.p. anhyd. 173–5°. Benzoyl: m.p. 169°.

3-Bromo-d-camphor-8-sulphonate: prisms from EtOH or  $\text{H}_2\text{O}$ . M.p. 194°. Sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Almost insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether.  $[\alpha]_D + 58.8^\circ$  in  $\text{H}_2\text{O}$ , +  $50.3^\circ$  in  $\text{CHCl}_3$ , +  $72.3^\circ$  in MeOH.

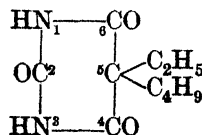
Benzoyl: needles from EtOH. M.p. 169°. Sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Mod. sol. EtOH,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ , pet. ether.

Picrate: yellow plates from EtOH. M.p. 195–6°. Spar. sol.  $\text{H}_2\text{O}$ .

Harris, *J. Chem. Soc.*, 1919, 115, 61.

Tattersall, Kipping, *J. Chem. Soc.*, 1903, 83, 825.

**Neonal** (Someryl, 5-ethyl-5-n-butylbarbituric acid)



$\text{C}_{10}\text{H}_{16}\text{O}_3\text{N}_2$

MW, 212

Cryst. from EtOH.Aq. M.p. 127–8°. Sol. EtOH, dil. alkalis. Spar. sol.  $\text{H}_2\text{O}$ .

N-p-Nitrobenzyl: m.p. 146°.

Hargreaves, Nixon, *Chem. Zentr.*, 1934, I, 2324.

Dox, Yoder, *J. Am. Chem. Soc.*, 1922, 44, 1580.

Poulenc, D.R.P., 481,129, (*Chem. Abstracts*, 1929, 23, 4950).

**Neonicotine.**

See under Anabesine.

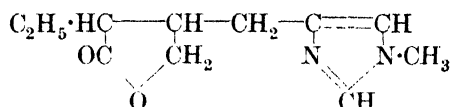
**Neopentane.**

See Tetramethylmethane.

**Neopentyl Alcohol.**

tert.-Butylcarbinol, *q.v.*

**Neopilcarpine** (*Methylpilocarpidine*)



$\text{C}_{11}\text{H}_{16}\text{O}_2\text{N}_2$

MW, 208

Cryst. M.p. 39–40°. Sol.  $\text{H}_2\text{O}$ , most org. solvents. Insol. ligroin.

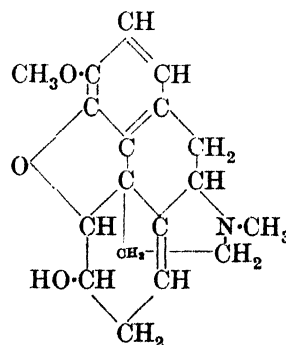
B.HCl: prisms from EtOH– $\text{Me}_2\text{CO}$ . M.p. 177°. Sol.  $\text{H}_2\text{O}$ , EtOH.  $[\alpha]_D + 66.4^\circ$ . De-liquescence.

B.HNO<sub>3</sub>: needles from EtOH. M.p. 94–5°.

Picrate: needles from EtOH. M.p. 117–19°.

Burtles, Lee, Pyman, *J. Chem. Soc.*, 1925, 127, 581.

**Neopine** ( $\beta$ -Codeine)



$\text{C}_{18}\text{H}_{21}\text{O}_3\text{N}$

MW, 299

Alkaloid of opium. Colourless needles from pet. ether. M.p. 127–127.5°. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ .  $[\alpha]_D^{25} - 28.10^\circ$  in  $\text{CHCl}_3$ . Optically inactive in  $\text{H}_2\text{O}$ . Hydrogenation  $\rightarrow$  dihydrocodeine.

B.HBr: m.p. 282–3° decomp.  $[\alpha]_D^{25} + 17.32^\circ$  in  $\text{H}_2\text{O}$ .

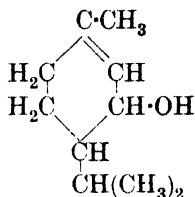
*Acetyl*: pale yellow varnish. *Methiodide*: long needles from MeOH. M.p. 256–7°.

Dobbie, Lauder, *J. Chem. Soc.*, 1911, 99, 34.

v. Duin, Robinson, Smith, *J. Chem. Soc.*, 1926, 903.

Gulland, Robinson, *J. Chem. Soc.*, 1923, 123, 980, 998.

### Neopiperitol ( $\Delta^1$ -p-Menthenol-3)



$C_{10}H_{18}O$

MW, 154

*d.*

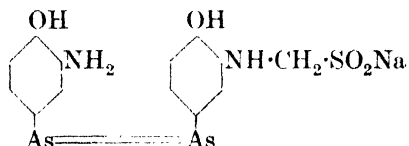
Liq. B.p. 96.5–98.5°/15.5 mm.  $D_4^{25}$  0.9119.  $n_D^{17}$  1.4976,  $n_D^{25}$  1.4729.  $[\alpha]_D^{16} + 21.22^\circ$ .

*dl.*

Viscous liq. with characteristic odour. B.p. 94–6°/15.5 mm.  $n_D^{19.6}$  1.4740.

Read, Storey, *J. Chem. Soc.*, 1930, 2779.

### Neosalvarsan (914, Novarsenobenzene)



$C_{13}H_{13}O_4N_2SAs_2Na$

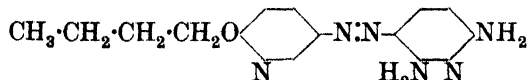
MW, 466

Yellow solid. Sol.  $H_2O \rightarrow$  a neutral solution. Solutions decomp. easily by heat, light or exposure to air. Employed in treatment of protozoal diseases.

M.L.B., D.R.P., 245,746, (*Chem. Zentr.*, 1912, I, 1522); D.R.P., 260,235, (*Chem. Zentr.*, 1913, II, 105).

Dyke, King, *J. Chem. Soc.*, 1934, 1707.

### Neotropine (2 : 6-Diamino-6'-butyloxy-3 : 3'-azopyridine)



$C_{14}H_{18}ON_6$

MW, 286

Orange cryst. M.p. 129°. Used as anti-septic.

*Hydrochloride*: m.p. 227°.

*Picrate*: m.p. 240° decomp.

*Picrolonate*: m.p. 204° decomp.

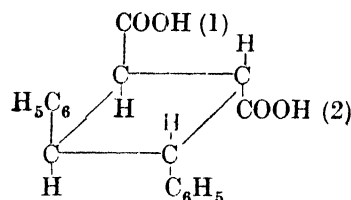
Zernik, *Pharm. Ztg.*, 1930, 75, 1204,

(*Chem. Zentr.*, 1930, II, 3812).

Schering-Kahlbaum, E.P., 341,598,

(*Chem. Zentr.*, 1931, I, 2678).

### Neotruxinic Acid



$C_{18}H_{16}O_4$

MW, 296

*d.*

Fine needles from EtOH.Aq. M.p. 236–7°.  $[\alpha]_D^{19} + 52.63^\circ$  in  $Me_2CO$ .

*Cinchonine salt*: cryst. from EtOH. M.p. 216–17°.

*Di-Me ester*:  $C_{20}H_{20}O_4$ . MW, 324. Cryst. from MeOH. M.p. 100°.  $[\alpha]_D^{20} + 48.11^\circ$  in  $Me_2CO$ .

*Di-Et ester*:  $C_{22}H_{24}O_4$ . MW, 352. Needles from EtOH. M.p. 53°.  $[\alpha]_D^{20} + 18.33^\circ$  in  $Me_2CO$ .

*Dichloride*:  $C_{18}H_{14}O_2Cl_2$ . MW, 333. Cryst. from  $C_6H_6$ -pet. ether. M.p. 103–4°.  $[\alpha]_D^{20} - 15.98^\circ$  in  $Me_2CO$ .

*Diamide*:  $C_{18}H_{18}O_2N_2$ . MW, 294. Cryst. from EtOH. M.p. 260–1°. Spar. sol. most solvents.

*Dianilide*: cryst. from hot AcOH. M.p. 226–7°.  $[\alpha]_D^{17} - 52.23^\circ$  in  $Me_2CO$ . Spar. sol. most solvents.

*l.*

Cryst. from EtOH. M.p. 236–7°.  $[\alpha]_D^{19} - 53.95^\circ$  in  $Me_2CO$ .

*Quinine salt*: felted cryst. from EtOH. M.p. 138°.

*Di-Me ester*: cryst. from EtOH. M.p. 100–1°.  $[\alpha]_D^{20} - 51.99^\circ$  in  $Me_2CO$ .

*Methylamide*: needles from MeOH.Aq. M.p. 126–7°.

*Ethylamide*: needles from EtOH.Aq. M.p. 175°.  $[\alpha]_D^{19} + 30.30^\circ$  in  $Me_2CO$ .

*dl.*

Fine needles from  $C_6H_6$ -AcOH. M.p. 209–209.5°. Sol. EtOH,  $Et_2O$ , AcOH. Spar. sol.  $C_6H_6$ . KOH fusion  $\rightarrow$   $\delta$ -truxinic acid.  $Ac_2O$  at 160°  $\rightarrow$   $\beta$ -truxinic acid.

$NH_4$  salt: cryst. powder. M.p. 235°. Sol.  $H_2O$ . Insol. EtOH. Above m.p.  $\rightarrow$   $\beta$ -truxinic imide.



Cryst. powder from EtOH. M.p. 42.5–43° (40–1°). Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Melt over HNO<sub>3</sub> → *trans*-14-tetracosenic acid.

Na salt: sol. hot EtOH.

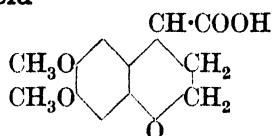
Pb salt: spar. sol. cold EtOH.

Klenk, *Z. physiol. Chem.*, 1926, **157**, 283.

Tsujimoto, *J. Soc. Chem. Ind. Japan*, 1927, **30**, 868.

Hale, Lycan, Adams, *J. Am. Chem. Soc.*, 1930, **52**, 4536.

### Netoric Acid



C<sub>12</sub>H<sub>14</sub>O<sub>5</sub> MW, 238

Cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 91–2°, anhyd. 134°. Does not reduce Fehling's.

Me ester: C<sub>13</sub>H<sub>16</sub>O<sub>5</sub>. MW, 252. Needles. M.p. 60°.

Robertson, Rusby, *J. Chem. Soc.*, 1936, 212.

Smith, La Forge, *J. Am. Chem. Soc.*, 1930, **52**, 4596.

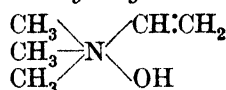
Clark, *J. Am. Chem. Soc.*, 1932, **54**, 2538.

Takei, Miyajima, Ono, *Ber.*, 1932, **65**, 289.

### Neuridine.

See Spermine.

Neurine (Trimethylvinylammonium hydroxide)



C<sub>5</sub>H<sub>13</sub>ON MW, 103

Occurs free and combined in many animal and vegetable products, e.g. bile, brain, yolk of egg, etc.

Liq. Forms cryst. hydrate with 3 mols. H<sub>2</sub>O. Easily decomp. → (CH<sub>3</sub>)<sub>3</sub>N. Dist. → (CH<sub>3</sub>)<sub>3</sub>N + CH:CH<sub>2</sub>OH. Very poisonous.

Bromide: plates from EtOH–Et<sub>2</sub>O. M.p. 194° decomp. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

Iodide: needles. M.p. 196°. Sol. H<sub>2</sub>O, EtOH.

Aurichloride: long yellow needles. M.p. 248° decomp.

Chloroplatinate: m.p. 213–14° (195.5–198°) decomp.

Picrate: golden-yellow needles from H<sub>2</sub>O. M.p. 246°. Sol. H<sub>2</sub>O, hot EtOH. Spar. sol. CHCl<sub>3</sub>, cold EtOH.

Hofmann, *Compt. rend.*, 1858, **47**, 559.

Meyer, Hopff, *Ber.*, 1921, **54**, 2277.

Renshaw, Ware, *J. Am. Chem. Soc.*, 1925, **47**, 2992.

### Neuronal (1-Bromodiethylacetamide)



C<sub>6</sub>H<sub>12</sub>ONBr MW, 194

Cryst. M.p. 66–7°. Sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Less sol. pet. ether. Sol. 115 parts cold H<sub>2</sub>O. Hypnotic and sedative.

Kalle, D.R.P., 170,629, (*Chem. Zentr.*, 1906, I, 1807); D.R.P., 158,220, (*Chem. Zentr.*, 1905, I, 635).

### Nevile and Winthers' Acid.

See 1-Naphthol-4-sulphonic Acid.

### Ngaïol

C<sub>15</sub>H<sub>24</sub>O<sub>3</sub> MW 252

Constituent of ngaïo tree. Exists in two isomeric forms. (i) B.p. 191–2°/29 mm. D<sub>20</sub><sup>20</sup> 1.0163. n<sub>D</sub><sup>20</sup> 1.4784. [α]<sub>D</sub> –25.00°. (ii) B.p. 188–90°/29 mm. D<sub>20</sub><sup>20</sup> 1.013. n<sub>D</sub><sup>20</sup> 1.4794.

Me ether: C<sub>16</sub>H<sub>26</sub>O<sub>3</sub>. MW, 266. B.p. 178–9°/29 mm. D<sub>20</sub><sup>20</sup> 0.9913. n<sub>D</sub><sup>20</sup> 1.4701. [α]<sub>D</sub> –16.82°.

Acetyl deriv.: yellow oil. B.p. 190–2°/29 mm. D<sub>20</sub><sup>20</sup> 1.0337. n<sub>D</sub><sup>20</sup> 1.4720.

McDowall, *J. Chem. Soc.*, 1925, **127**, 2205; 1928, 1324.

### Ngaïone

C<sub>15</sub>H<sub>22</sub>O<sub>3</sub> MW, 250

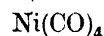
Liq. B.p. 183°/27 mm.

Semicarbazone: m.p. 120–2° decomp.

p-Nitrophenylhydrazone: m.p. 103°.

McDowall, *J. Chem. Soc.*, 1925, **127**, 2200.

### Nickel carbonyl (Nickel tetracarbonyl)



C<sub>4</sub>O<sub>4</sub>Ni MW, 170.5

Yellow liq. B.p. 43.2–43.33°/769 mm. Crit. temp. 200°. Crit. press. 30 atmospheres. Vapour explodes at 60° but not in presence of an inert gas. Halogens → nickel halides. H<sub>2</sub>S → NiS. C<sub>6</sub>H<sub>6</sub> + AlCl<sub>3</sub> → anthracene. Used as antidetonant in motor fuels.

v. Duin, *Rec. trav. chim.*, 1927, **46**, 381.

I.G., E.P., 394,906, (*Chem. Abstracts*, 1934, **28**, 267).

Manchot, Gall, *Ber.*, 1929, **62**, 678.

Dewar, Jones, *Proc. Roy. Soc.*, 1903, **71**, 427; *J. Chem. Soc.*, 1904, **85**, 203, 212.

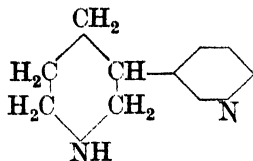
### Nickel tetracarbonyl.

See Nickel carbonyl.

### Nicotine.

Recent work has shown this substance to be a mixture of *l*-anabasine and nornicotine, *q.v.*

**Nicotidine** (3-[3-Pyridyl]-piperidine, 3-[3-piperidyl]-pyridine)



$C_{10}H_{14}N_2$

MW, 162

Viscous, pale yellow oil. B.p. 287–9° (284–5°). Very sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ . Poisonous.

*Picrate*: cryst. from  $H_2O$ . M.p. 206°.

Smith, *J. Am. Chem. Soc.*, 1931, **53**, 282.

Skraup, Vortmann, *Monatsh.*, 1883, **4**, 597.

### Nicotimine

$C_{10}H_{14}N_2$

MW, 162

Alkaloid found in *Nicotiana tabacum*. Oil. B.p. 250–5°. Misc. with  $H_2O$ , org. solvents in all proportions. Reacts alkaline.

*Hydrochloride*: cryst. Very deliquescent.

*Chloroplatinate*: bright yellow cryst. Decomp. at 270°.

*Aurichloride*: bright yellow leaflets. M.p. 182–5° decomp.

*Mercurichloride*: needles. Decomp. at 190°.

*Picrate*: yellow prisms. M.p. 163°.

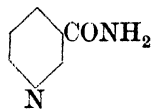
Pictet, Rotschy, *Ber.*, 1904, **37**, 1225.

Pictet, *Arch. Pharm.*, 1906, **244**, 388.

Ehrenstein, *Arch. Pharm.*, 1931, **269**, 627.

Cf. Smith, *J. Am. Chem. Soc.*, 1931, **53**, 278.

### Nicotinamide (Nicotinic acid amide)



$C_6H_6ON_2$

MW, 122

Needles from  $C_6H_6$ . M.p. 122°. Dist. with  $P_2O_5$  at 25 mm. press., or  $SOCl_2$  at 100° → 3-cyanopyridine.

*N-Me*:  $C_7H_8ON_2$ . MW, 136. Needles from  $C_6H_6$  or  $CHCl_3$ -ligroin. M.p. 104–5°.

*N-Di-Et*: coramine.  $C_{10}H_{14}ON_2$ . MW, 178. Yellowish oil. B.p. 280°, 175°/25 mm. Sol.  $H_2O$ , org. solvents. Used as an analeptic.

*N-Dipropyl*:  $C_{12}H_{18}ON_2$ . MW, 206. Yellowish oil. B.p. 184°/17 mm.

*N-Isoamyl*:  $C_{11}H_{16}ON_2$ . MW, 192. Thick liq. B.p. 191°/8 mm. Sol.  $Et_2O$ . Easily decomp.

*Chloroaurate*: m.p. 205°.

Pollak, *Monatsh.*, 1895, **16**, 53.

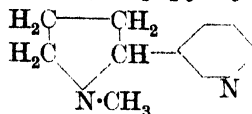
Camps, *Arch. Pharm.*, 1902, **240**, 354.

Pictet, Sussdorff, *Chem. Zentr.*, 1898, **I**, 677.

Hartmann, Seiberth, U.S.P., 1,403,117, (*Chem. Abstracts*, 1922, **16**, 935).

La Forge, *J. Am. Chem. Soc.*, 1928, **50**, 2480.

### Nicotine (1-Methyl-2-[3-pyridyl]-pyrrolidine)



$C_{10}H_{14}N_2$

MW, 162

*l.*

Alkaloid from *Nicotiana tabacum*. Liq. with odour of Py. B.p. 246.1°/730.5 mm.  $D_4^{20}$  1.0097.  $n_D^{20}$  1.5280.  $[\alpha]_D^{20}$  –166.39° to –168.5°. Very sol. EtOH,  $Et_2O$ , pet. ether. Misc. with  $H_2O$  below 60° and above 210°. Volatile in steam. Readily turns brown in air.  $HgCl_2$  gives white cryst. ppt. Aq.  $K_2PtI_6$  → black ppt.

*B.HCl*:  $[\alpha]_D + 102.2°$ .

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*:  $[\alpha]_D + 84.8°$ .

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: decomp. at 275°.

*Acetate*:  $[\alpha]_D + 110.29°$ .

*d-Acid tartrate*: cryst. + 2 $H_2O$  from EtOH- $Et_2O$ . M.p. 88–9°.  $[\alpha]_D^{27} + 26.60°$  in  $H_2O$ .

*d-Ditartrate*: cryst. + 2 $H_2O$  from EtOH- $Et_2O$ . M.p. 68–5°.  $[\alpha]_D^{29.5} + 25.99°$  in  $H_2O$ .

*Picrate*: prisms from EtOH. M.p. 218°.

*Picrolonate*: cryst. from EtOH. M.p. 238° (213°).

*Tetrachloroiodide*: orange cryst. M.p. 150°. Very stable.

*d.*

Liq. B.p. 245.5–246.5°/729 mm.  $D_4^{20}$  1.0171.  $D_4^{20}$  1.0094.  $[\alpha]_D^{20} + 163.17°$ .

*l-Ditartrate*: cryst. from  $H_2O$ . M.p. 88–9°.  $[\alpha]_D^{15} - 25.58°$  in  $H_2O$ .

*dl.*

Tetrahydronicotyrine. Liq. B.p. 242.3°.  $D_4^{20}$  1.0082.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: decomp. about 280°.

*Picrate*: long yellow needles from  $H_2O$ . M.p. 218°.

*Methiodide*: cryst. from MeOH. M.p. 219°.

Laiblin, *Ann.*, 1879, **196**, 130.

Pictet, Rotschy, *Ber.*, 1901, **34**, 696; 1904, **37**, 1226.

Späth, Bretschneider, *Ber.*, 1928, **61**, 327.

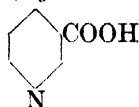
Craig, *J. Am. Chem. Soc.*, 1933, **55**, 2856.

Späth, Kuffner, *Ber.*, 1935, **68**, 494.

$\alpha$ -Nicotine.

See under 2-[2-Pyridyl]-pyrrolidine.

**Nicotinic Acid** (*Pyridine-3-carboxylic acid*)



$C_6H_5O_2N$  MW, 123  
Needles from  $H_2O$  or EtOH. M.p.  $232^\circ$ .  
Sol. hot  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ . Sublimes.  
 $k = 1.4 \times 10^{-5}$  at  $25^\circ$ .

$B, HCl$ : prisms or plates from  $H_2O$ . M.p.  $274^\circ$ .

$B, HBr$ : plates. M.p.  $275^\circ$ . Sublimes.

$B, HNO_3$ : plates or prisms +  $H_2O$ . M.p.  $190-2^\circ$  ( $185^\circ$ ).

$B, HAuCl_4$ : yellow leaflets. M.p.  $207^\circ$ .

*Piperidine salt*: needles. M.p.  $122^\circ$ .

*Tetrachloriodide*: yellow. M.p.  $137^\circ$ .

*Ethochloroplatinate*: light yellow needles from EtOH-HCl. M.p.  $205^\circ$  decomp. Spar. sol.  $H_2O$ .

*Me ester*:  $C_7H_7O_2N$ . MW, 137. Cryst. M.p.  $38^\circ$ . B.p.  $204^\circ$ . Sol.  $H_2O$ , EtOH,  $C_6H_6$ .

*Et ester*:  $C_8H_9O_2N$ . MW, 151. Liq. B.p.  $223-4^\circ$ ,  $107-8^\circ/17$  mm.,  $103^\circ/5$  mm. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin.  $B, HCl$ , needles. M.p.  $126-7^\circ$ .  $B, HNO_3$ : m.p.  $185^\circ$ .  $B, HAuCl_4$ : pale yellow leaflets from EtOH-HCl. M.p.  $117^\circ$ .

$B_2, H_2PtCl_6$ : yellow needles from EtOH. M.p.  $161^\circ$ . *Ethochloroplatinate*: yellow plates from EtOH-HCl. M.p.  $176^\circ$ . Spar. sol.  $H_2O$ . *Ethochloroaurate*: leaflets from EtOH-Aq. M.p.  $59^\circ$ .

*Propyl ester*:  $C_9H_{11}O_2N$ . MW, 165. Liq. B.p.  $232^\circ$ .

*Isoamyl ester*:  $C_{11}H_{15}O_2N$ . MW, 193. Liq. B.p.  $259^\circ$ .

*Phenyl ester*:  $C_{12}H_9O_2N$ . MW, 199. Cryst. from EtOH. M.p.  $71^\circ$ .

*Chloride*: see Nicotinyl chloride.

*Amide*: see Nicotinamide.

*Nitrile*: see 3-Cyanopyridine.

*Anilide*: needles +  $2H_2O$  from  $H_2O$ , m.p.  $85^\circ$ : needles from  $C_6H_6$ -ligroin or  $CHCl_3$ -ligroin, m.p.  $132^\circ$ . Sol. hot  $H_2O$ , EtOH,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ , ligroin.

*p-Toluidide*: needles from boiling  $H_2O$ . M.p.  $150^\circ$ .

*Methylbetaine*: see Trigonelline.

*Ethylbetaine*: plates. M.p.  $84-6^\circ$ . Sol.  $H_2O$ . Less sol. EtOH. Insol.  $Et_2O$ , usual solvents.

*Anhydride*: cryst. from  $C_6H_6$ . M.p.  $122-4^\circ$ . Hygroscopic.

*Hydrazide*: needles from EtOH or  $C_6H_6$ . M.p.  $158-9^\circ$ . Sol.  $H_2O$ , EtOH. Less sol.  $C_6H_6$ .  $B_2HCl$ : needles from alc. HCl +  $Et_2O$ . M.p.  $227^\circ$ . *Benzylidene*: cryst. from  $C_6H_6$ . M.p.  $149-52^\circ$ .

*Azide*: cryst. M.p.  $47-8^\circ$ .

*Phenylhydrazide*: m.p.  $185^\circ$ .

Graf, *Biochem. Z.*, 1930, **229**, 166.

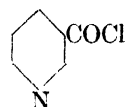
Engler, *Ber.*, 1894, **27**, 1787.

Pictet, Sussdorff, *Chem. Zentr.*, 1898, **I**, 677.

Camps, *Arch. Pharm.*, 1902, **240**, 353.

Pollak, *Monatsh.*, 1895, **16**, 46.

McElvain, *Organic Syntheses*, Collective Vol. **I**, 378.

**Nicotinyl chloride** (*Nicotinic acid chloride*)

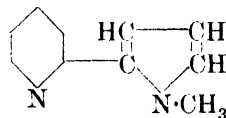
$C_6H_4ONCl$  MW, 141.5

Cryst. M.p.  $15-16^\circ$ . B.p.  $85^\circ/12$  mm.

$B, HCl$ : needles. M.p.  $155.5-156.5^\circ$ . Sol.  $H_2O$ . Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , pet. ether. Sublimes.

Meyer, Graf, *Ber.*, 1928, **61**, 2205.

Späth, Spitzer, *Ber.*, 1926, **59**, 1479.

**2 : 2'-Nicotyrine** ( $\alpha$ -Nicotyrine, *N-methyl-2-[2-pyridyl]-pyrrole*)

$C_{10}H_{10}N_2$  MW, 158

Exists in two forms.

(i) Liq. F.p.  $-28^\circ$ . B.p.  $273^\circ/764$  mm.,  $149-50^\circ/22$  mm. Turns red on standing. Gives blue col. with pine splint + HCl. HCl + dimethylaminobenzaldehyde  $\rightarrow$  intense reddish-violet col.  $\rightarrow$  brownish-red on standing.

*Picrate*: yellow leaflets from EtOH. M.p.  $143^\circ$  ( $138-9^\circ$ ).

*Methiodide*: yellowish-brown cryst. from  $H_2O$ . M.p.  $188^\circ$ .

(ii) Cryst. M.p.  $43.5-44.5^\circ$ . Turns brown on standing. Sol. EtOH,  $C_6H_6$ , dil. HCl. Mod. sol.  $Et_2O$ . Insol. cold  $H_2O$ . HCl + dimethylaminobenzaldehyde  $\rightarrow$  intense reddish-violet col.  $\rightarrow$  bluish-violet on standing.

*Picrate*: m.p.  $193-4^\circ$ .

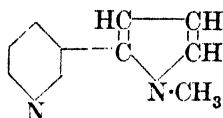
*Methiodide*: needles from EtOH. M.p.  $146.2-146.6^\circ$ . Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .

Tschitschibabin, Bylinkin, *Ber.*, 1923, **56**, 1748.

Wibaut, Dingemans, *Rec. trav. chim.*, 1923, **42**, 1033.

Wibaut, Coppens, *Rec. trav. chim.*, 1924, **43**, 526.

**3 : 2'-Nicotyrine** (N-Methyl-2-[3-pyridyl]-pyrrole)



$C_{10}H_{10}N_2$

MW, 158

Liq. B.p. 280–1°/744 mm., 150°/15 mm.  $D_{13}^{20}$  1.124. Sol. EtOH. Spar. sol.  $H_2O$ .

$B_2H_2PtCl_6$ : reddish-brown plates +  $2H_2O$  from  $H_2O$ . M.p. 158–60°.

Picrate: m.p. 168–9° (163–4°).

Wibaut, Overhoff, *Rec. trav. chim.*, 1928, 47, 925.

Blau, *Ber.*, 1894, 27, 2537.

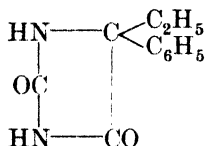
### Nipecotic Acid.

See Hexahydronicotinic Acid.

### Niperyt.

See under Pentaerythritol.

**Nirvanol** (5-Ethyl-5-phenylhydantoin)



$C_{11}H_{12}O_2N_2$

MW, 204

*d*-.

Flaky platelets from 10% EtOH. M.p. 237°.  $[\alpha]_D^{20}$  + 123° in EtOH, + 169° in aq. alkalis. Slightly less effective than *dl*-, but only one third as toxic.

*l*-.

Cryst. from EtOH.Aq. M.p. 235–7°.  $[\alpha]_D^{20}$  – 121° in EtOH, – 167° in aq. alkalis.

*dl*-.

Prisms from EtOH.Aq. M.p. 199–200° (197°). Sol. warm  $Me_2CO$ , EtOH, AcOH. Sol. 1650 parts cold, 110 parts hot  $H_2O$ . Insol.  $C_6H_6$ . Used as hypnotic.

Sobotka, Holzman, Kahn, *J. Am. Chem. Soc.*, 1932, 54, 4698.

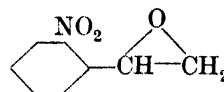
M.L.B., Swiss P., 72,561, (*Chem. Abstracts*, 1917, 11, 186); 73,891, (*Chem. Abstracts*, 1917, 11, 1259).

Hermanns, U.S.P., 1,285,703, (*Chem. Abstracts*, 1919, 13, 248).

v. Heyden, D.R.P., 335,993, (*Chem. Abstracts*, 1923, 17, 1802).

Bergs, D.R.P., 566,094, (*Chem. Abstracts*, 1933, 27, 1001).

**Nitraldin** (o-Nitrophenylethylene oxide)



$C_8H_7O_3N$

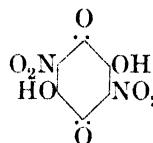
MW, 165

Pale yellow plates from MeOH. M.p. 65°. B.p. 150°/15 mm., 144°/10 mm. Sol.  $Et_2O$ ,  $C_6H_6$ . Less sol. MeOH. Mod. sol. hot pet. ether. Spar. sol. hot  $H_2O$ . Decomp. vigorously above 200°. Deflagrates violently on contact with conc.  $H_2SO_4$ .

Arndt, Partale, *Ber.*, 1927, 60, 451.

Arndt, Eistert, Partale, *Ber.*, 1928, 61, 1107.

**Nitrannilic Acid** (3 : 6-Dinitro-2 : 5-dihydroxy-p-benzoquinone)



$C_6H_2O_8N_2$

MW, 230

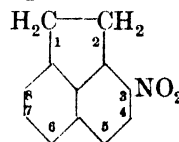
Golden-yellow plates +  $H_2O$ . M.p. 86–7° (100°) decomp. Very sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ . Explodes on heating in sealed tube.

Meyer, *Ber.*, 1924, 57, 326.

### Nitroacetaldoxime.

See Methazonic Acid.

### 3-Nitroacenaphthene



$C_{12}H_9O_2N$

MW, 199

Yellow needles from AcOH. M.p. 151.5°.

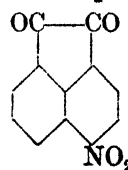
Morgan, Harrison, *J. Soc. Chem. Ind.*, 1930, 49, 413r.

### 5-Nitroacenaphthene.

Yellow needles from ligroin. M.p. 106° (101°). Sol. hot  $H_2O$ , EtOH,  $Et_2O$ , ligroin. Conc.  $H_2SO_4$  → bluish-violet col.

Sachs, Mosebach, *Ber.*, 1911, 44, 2854.

### 5-Nitroacenaphthenequinone



$C_{12}H_5O_4N$

MW, 227

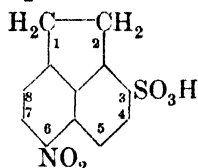
Yellow needles from AcOH. M.p. 218° (199°).  
*Monophenylhydrazone*: reddish-brown needles from Py. M.p. 234–5° (186°).

*Diphenylhydrazone*: m.p. 148°.

Rowe, Davies, *J. Chem. Soc.*, 1920, 117, 1349.

Mayer, Kauffmann, *Ber.*, 1920, 53, 296.

### 6-Nitroacenaphthene-3-sulphonic Acid



$C_{12}H_9O_5NS$

MW, 279

Yellow amorph. powder.

*Me ester*:  $C_{13}H_{11}O_5NS$ . MW, 293. Needles from ligroin. M.p. 143–4°.

*Chloride*:  $C_{12}H_8O_4NCIS$ . MW, 297.5. Reddish-brown amorph. powder. Sol.  $C_6H_6$ . Spar. sol. ligroin.

Dziewoński, Orzelski, *Chem. Zentr.*, 1927, I, 1461.

### 6-Nitroacenaphthene-4-sulphonic Acid.

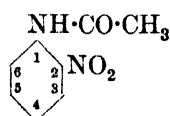
Yellow needles.

*Me ester*: yellow needles from ligroin. M.p. 146°.

*Chloride*: yellow cryst. from AcOH. Very sol.  $C_6H_6$ .

See previous reference.

### o-Nitroacetanilide



$C_8H_8O_3N_2$

MW, 180

Yellow plates or prisms from ligroin. M.p. 94°. Sol. hot  $H_2O$ , EtOH,  $CHCl_3$ .  $D^{15}_D$  1.419.

*N-Me*: see under 2-Nitro-*N*-methylaniline.

Böeseken, *Rec. trav. chim.*, 1912, 31, 351.

Menke, E.P., 235,698, (*Chem. Abstracts*, 1926, 20, 916).

### m-Nitroacetanilide.

Leaflets from EtOH. M.p. 154–6°. Sol.  $PhNO_2$ . Mod. sol.  $CHCl_3$ .

*N-Me*: see under 3-Nitro-*N*-methylaniline.

*N-Et*: see under 3-Nitro-*N*-ethylaniline.

Kauffmann, *Ber.*, 1909, 42, 3482.

Pawlewski, *Ber.*, 1898, 31, 661.

### p-Nitroacetanilide.

Prisms. M.p. 215–16° (207°). Heat of comb.

$C_p$  969 Cal. Sol. cold KOH with orange col. Spar. sol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ , ligroin.

*N-Me*: see under 4-Nitro-*N*-methylaniline.

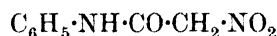
*N-Et*: see under 4-Nitro-*N*-ethylaniline.

*N-Benzoyl*: plates from AcOEt. M.p. 180°.

See first reference above and also

Witt, Utermann, *Ber.*, 1906, 39, 3903.

### ω-Nitroacetanilide



$C_8H_8O_3N_2$

MW, 180

Yellow plates from  $H_2O$ . M.p. 138–9°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOEt. Spar. sol. cold  $H_2O$ . Sol.  $Na_2CO_3$ . Red.  $\rightarrow$  glycine.

Steinkopf, Daege, *Ber.*, 1911, 44, 499.

### Nitroacetanilide.

See under Nitroanisidine.

### Nitroacetic Acid



$C_2H_3O_4N$

MW, 105

Needles from  $CHCl_3$ . M.p. 87–9° decomp. Very sol. EtOH,  $Et_2O$ . Sol.  $C_6H_6$ , toluene, hot  $CHCl_3$ . Insol. pet. ether. Heated with  $H_2O \rightarrow$  nitromethane.

*Me ester*:  $C_3H_5O_4N$ . MW, 119. Liq. B.p. 107°/28 mm., 94–5°/16 mm.  $D^{15}_D$  1.320. Spar. sol.  $H_2O$ .

*Et ester*:  $C_4H_7O_4N$ . MW, 133. Liq. B.p. 105–7°/25 mm., 93–5°/10 mm. Misc. with EtOH. Spar. sol.  $H_2O$ .  $D^{15}_D$  1.226,  $D^{20}_D$  1.992.  $k = 1.4 \times 10^{-6}$  at 25°. Aq. sol. reacts acid to litmus. *NH<sub>4</sub> salt*: needles from EtOH. M.p. 124°.

*Propyl ester*:  $C_5H_9O_4N$ . MW, 147. B.p. 105°/18 mm.

*Isopropyl ester*: b.p. 92–3°/12 mm. *NH<sub>4</sub> salt*: m.p. 107–9°.

*Isobutyl ester*:  $C_6H_{11}O_4N$ . MW, 161. B.p. 111.5–112.5°/15 mm., 102°/8 mm. *NH<sub>4</sub> salt*: m.p. 110–12°.

*Isoamyl ester*:  $C_7H_{13}O_4N$ . MW, 175. B.p. 122–3°/16 mm. *NH<sub>4</sub> salt*: m.p. 112–15°.

*Amide*:  $C_2H_4O_3N_2$ . MW, 104. Needles from  $C_6H_6$ . M.p. 106–7°. Sol.  $H_2O$ , EtOH,  $Me_2CO$ . Spar. sol.  $C_6H_6$ . Insol. ligroin.

*Nitrile*:  $C_2H_2O_2N_2$ . MW, 86. Yellow oil. Decomp. on heating.  $D^{18}$  1.36. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ .

Bouveault, Wahl, *Bull. soc. chim.*, 1904, 31, 851.

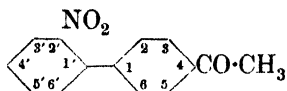
Steinkopf, *Ber.*, 1909, 42, 619, 3925.

Steinkopf, Haugen, Schkade, *Ann.*, 1923, 434, 26.



**Nitroacetnaphthalide.**

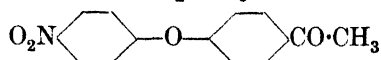
See under Nitronaphthylamine.

**2'-Nitro-4-acetodiphenyl** $C_{14}H_{11}O_3N$ 

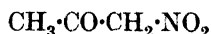
MW, 241

Prisms from EtOH. M.p. 110°.  $KMnO_4 \rightarrow$  2'-nitrodiphenyl-4-carboxylic acid.Grieve, Hey, *J. Chem. Soc.*, 1933, 970.**4'-Nitro-4-acetodiphenyl.**Yellow prisms or needles from EtOH or  $C_6H_6$ . M.p. 152-3°.  $KMnO_4 \rightarrow$  4'-nitrodiphenyl-4-carboxylic acid.

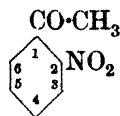
See previous reference and also

Diltthey, Neuhaus, Reis, Schommer, *J. prakt. Chem.*, 1930, 124, 124.I.G., F.P., 735,846, (*Chem. Abstracts*, 1933, 27, 1001).**4'-Nitro-4-acetodiphenyl Ether** $C_{14}H_{11}O_4N$ 

MW, 257

Needles from 95% EtOH. M.p. 82-3° (80-1°). Conc.  $H_2SO_4 \rightarrow$  greenish-yellow col.Suker, Oberg, *J. Am. Chem. Soc.*, 1931, 53, 1567.Diltthey, Bach, Grütering, Hausdörfer, *J. prakt. Chem.*, 1927, 117, 361.**Nitroacetone** $C_3H_5O_3N$ 

MW, 103

Plates or needles from  $Et_2O$  or  $C_6H_6$ . M.p. 49-50°. B.p. 103-4°/24 mm. Very sol.  $C_6H_6$ . Sol. EtOH,  $Et_2O$ . Mod. sol.  $H_2O$ . Reacts acid. Gives no col. with  $FeCl_3$ .*Oxime*: oil. Sol.  $H_2O$ .*Semicarbazone*: needles from EtOH. M.p. 163-4°.*Anil*: yellow needles from EtOH or ligroin. M.p. 87°.Harries, *Ann.*, 1901, 319, 251. **$\omega$ -Nitroacetophenone** $C_8H_7O_3N$ 

MW, 165

Oil. B.p. 178-9°/32 mm., 159°/16 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $H_2O$ .Kermack, Smith, *J. Chem. Soc.*, 1929, 814.***m*-Nitroacetophenone.**

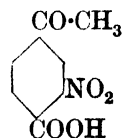
Needles from EtOH. M.p. 81°. B.p. 202°, 167°/18 mm. Volatile in steam. Easily reduced.

*Oxime*: needles from boiling  $H_2O$ . M.p. 131-2°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH. Mod. sol.  $C_6H_6$ . Spar. sol.  $CS_2$ , pet. ether. *Methylether*: needles. M.p. 63-4°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH,  $CS_2$ . Mod. sol. EtOH, pet. ether. Volatile in steam.*Phenylhydrazones*: orange needles from EtOH. M.p. 128°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol. ligroin.*o*-Tolylhydrazones: m.p. 135-6°.Gabriel, *Ber.*, 1882, 15, 3063.Corson, Hazen, *Organic Syntheses*, 1930, 10, 74.Morgan, Watson, *J. Soc. Chem. Ind.*, 1936, 55, 29T.***p*-Nitroacetophenone.**

Yellow prisms. M.p. 80-1°.

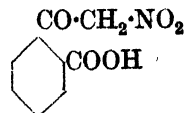
*Phenylhydrazones*: cryst. from EtOH. M.p. 132°.Engler, Zielke, *Ber.*, 1889, 22, 203.Barkenbus, Clements, *J. Am. Chem. Soc.*, 1934, 56, 1369. **$\omega$ -Nitroacetophenone.**

See Benzoylnitromethane.

**3-Nitroacetophenone-4-carboxylic Acid**  
(2-Nitro-4-acetobenzoic acid) $C_9H_7O_5N$ 

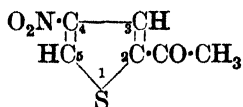
MW, 209

Pale yellow cryst. M.p. 178-9°.

*Methylester*:  $C_{10}H_9O_5N$ . MW, 223. Needles. M.p. 58°. B.p. 211-14°/22 mm.Mayer, Stark, Schön, *Ber.*, 1932, 65, 1335. **$\omega$ -Nitroacetophenone-2-carboxylic Acid**  
(2-Nitroacetobenzoic acid, *o*-carboxybenzoylnitromethane) $C_9H_7O_5N$ 

MW, 209

Plates from  $H_2O$  at 80°. M.p. 121-5°. Alkalis  $\rightarrow$  yellow sols. Conc.  $H_2SO_4 \rightarrow$  greenish-blue col.  $\rightarrow$  indigo blue on standing.Gabriel, *Ber.*, 1903, 36, 574.

**4-Nitro-2-acetothienone** (4-Nitro-2-acetylthiophene)

$C_6H_5O_3NS$  MW, 171

Needles from EtOH. M.p. 127°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

Oxime: needles from H<sub>2</sub>O. M.p. 129° (127°).

Rinkes, *Rec. trav. chim.*, 1933, **52**, 538.

Steinkopf, Jaffé, *Ann.*, 1917, **413**, 336.

**5-Nitro-2-acetothienone** (5-Nitro-2-acetylthiophene).

Platelets from EtOH. M.p. 106-7°.

Oxime: light yellow leaflets from EtOH. M.p. 189°.

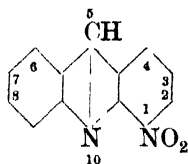
See previous references.

**Nitroacet-toluidide.**

See under Nitrotoluidine.

**4-Nitroacetylcatechol.**

See ω-Nitro-3:4-dihydroxyacetophenone.

**1-Nitroacridine**

$C_{13}H_8O_2N_2$  MW, 224

Plates from MeOH. M.p. 172° (167°). Sol. EtOH. Insol. H<sub>2</sub>O.

Jensen, Friedrich, *J. Am. Chem. Soc.*, 1927, **49**, 1051.

Lehmstedt, *Ber.*, 1927, **60**, 1371.

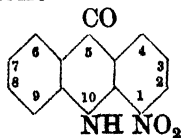
**3-Nitroacridine.**

Yellow plates from EtOH. M.p. 216°. Sublimes. Very sol. EtOH, Et<sub>2</sub>O. Mod. sol. CHCl<sub>3</sub>. Insol. H<sub>2</sub>O.

Jensen, Friedrich, *J. Am. Chem. Soc.*, 1927, **49**, 1049.

Lehmstedt, *Ber.*, 1927, **60**, 1370.

I.G., D.R.P., 545,265, (*Chem. Zentr.*, 1932, I, 2513).

**1-Nitroacridone**

$C_{13}H_8O_3N_2$  MW, 240

Orange needles from toluene. M.p. 262°. Sol. EtOH, AcOH, toluene, PhNO<sub>2</sub>.

Dict. of Org. Comp.—III.

N-Me:  $C_{14}H_{10}O_3N_2$ . MW, 254. Cryst. from EtOH. M.p. 168°. Mod. sol. EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. Et<sub>2</sub>O.

Ullmann, *Ann.*, 1907, **355**, 328.

Lehmstedt, *Ber.*, 1931, **64**, 2381.

Lehmstedt, Hundertmark, *ibid.*, 2386.

**2-Nitroacridone.**

Yellow needles. Does not melt below 350°. Sol. PhNO<sub>2</sub>, aniline, Py. Spar. sol. EtOH, AcOH, toluene. Insol. Et<sub>2</sub>O, ligroin.  $C_6H_5N(CH_3)_2 \rightarrow$  2-nitro-5-*p*-dimethylaminophenylacridine, m.p. 255°.

Ullmann, Wagner, *Ann.*, 1907, **355**, 364.

Lehmstedt, *Ber.*, 1932, **65**, 999.

**3-Nitroacridone.**

Yellow needles from PhNO<sub>2</sub>. Does not melt below 350°.  $C_6H_5N(CH_3)_2 \rightarrow$  3-nitro-5-*p*-dimethylaminophenylacridine, m.p. 225°.

N-Me: cryst. from AcOH. M.p. 276°. Mod. sol. common org. solvents. Insol. Et<sub>2</sub>O.

Ullmann, Bader, Labhardt, *Ber.*, 1907, **40**, 4797.

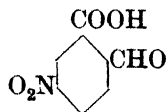
Lehmstedt, *Ber.*, 1927, **60**, 1371; 1931, **64**, 2381.

Lehmstedt, Hundertmark, *Ber.*, 1931, **64**, 2386.

**4-Nitroacridone.**

Yellow needles from PhNO<sub>2</sub>. Does not melt below 350°. Sol. PhNO<sub>2</sub>, aniline. Spar. sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.  $C_6H_5N(CH_3)_2 \rightarrow$  4-nitro-5-*p*-dimethylaminophenylacridine, m.p. 235°.

Ullmann, *Ann.*, 1907, **355**, 332.

**5-Nitro-2-aldehydobenzoic Acid** (5-Nitro-*o*-phthalaldehydic acid)

$C_8H_5O_5N$

MW, 195

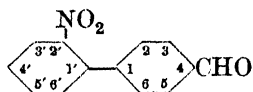
Cryst. M.p. 162°.

Phenyldiazone: bright red cryst. from MeOH or AcOH  $\rightarrow$  pyridazone deriv., m.p. 171°, on heating.

2:4-Dinitrophenylhydrazone: yellow cryst. M.p. 292°. Sol. MeOH, CHCl<sub>3</sub>, AcOH, AcOEt.

Borsche, Diacont, Hanau, *Ber.*, 1934, **67**, 680.

## 2'-Nitro-4-aldehydodiphenyl

 $C_{13}H_9O_3N$ 

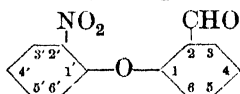
MW, 227

Yellow needles from EtOH. M.p. 101°.  $KMnO_4 \rightarrow$  2'-nitrodiphenyl-4-carboxylic acid. Grieve, Hey, *J. Chem. Soc.*, 1935, 114; 1933, 971.

## 4'-Nitro-4-aldehydodiphenyl.

Yellow needles from EtOH. M.p. 127-8°.  $KMnO_4 \rightarrow$  4'-nitrodiphenyl-4-carboxylic acid. See previous references.

## 2'-Nitro-2-aldehydodiphenyl Ether

 $C_{13}H_9O_4N$ 

MW, 243

Cryst. from ligroin. M.p. 77°.

Brewster, Strain, *J. Am. Chem. Soc.*, 1934, 56, 118.

## 4'-Nitro-2-aldehydodiphenyl Ether.

Cryst. from EtOH. M.p. 112°.

See previous reference.

## 2'-Nitro-4-aldehydodiphenyl Ether.

White powder. M.p. 84-5°.

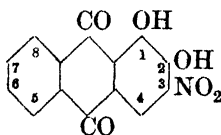
Suter, Oberg, *J. Am. Chem. Soc.*, 1931, 53, 1567.

## 4'-Nitro-4-aldehydodiphenyl Ether.

Needles from dil. AcOH. M.p. 104-5°.

See previous reference.

## 3-Nitroalizarin

 $C_{14}H_7O_6N$ 

MW, 285

Orange-yellow needles from  $C_6H_6$  or yellow plates from EtOH. M.p. 244°. Sol. EtOH,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Mod. sol.  $H_2O$ . Sol. conc.  $H_2SO_4$  with orange-yellow col.

*Diacetyl*: yellow needles from  $C_6H_6$ . M.p. 218°.

Schunck, Roemer, *Ber.*, 1879, 12, 584.

## 4-Nitroalizarin.

Golden-yellow needles from EtOH or AcOH. M.p. 289° decomp. Mod. sol. EtOH, AcOH.

Spar. sol.  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  deep yellow col.

*2-Me ether*:  $C_{15}H_9O_6N$ . MW, 299. Yellow plates from AcOH. M.p. 280-2°. Sol. Py,  $PhNO_2$ . Mod. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  orange-red sol.

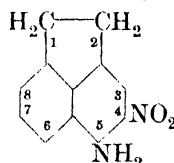
*Di-Me ether*:  $C_{16}H_{11}O_6N$ . MW, 313. Yellow needles from AcOH. M.p. 209-10°. Sol.  $Me_2CO$ ,  $CHCl_3$ , AcOH,  $PhNO_2$ . Spar. sol. EtOH,  $Et_2O$ ,  $CS_2$ . Conc.  $H_2SO_4 \rightarrow$  red sol.

*Diacetyl*: golden-yellow needles from AcOH. M.p. 194-195.5°.

M.L.B., D.R.P., 150,322, (*Chem. Zentr.*, 1904, I, 1043).

Brasch, *Ber.*, 1891, 24, 1611.

## 4-Nitro-5-aminoacenaphthene

 $C_{12}H_{10}O_2N_2$ 

MW, 214

Red prisms from EtOH. M.p. 222-4° (219°). Sol. 80 parts hot EtOH with greenish fluor. Sol. AcOH.

*Formyl*: yellow needles from AcOH. M.p. 227°. Sol. EtOH,  $CHCl_3$ .

*Acetyl*: golden-yellow needles from AcOH. M.p. 253° (251°). Sol. 60 parts hot EtOH. Spar. sol. other solvents. Conc.  $H_2SO_4 \rightarrow$  greenish-brown sol.

*Benzoyl*: pale yellow needles from AcOH. M.p. 233°.

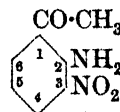
Sachs, Mosebach, *Ber.*, 1911, 44, 2857.

Morgan, Stanley, *J. Soc. Chem. Ind.*, 1924, 43, 343T.

## Nitroaminoacetanilide.

See under Nitrophenylenediamine.

## 3-Nitro-o-aminoacetophenone

 $C_8H_8O_3N_2$ 

MW, 180

Yellow needles from EtOH. M.p. 92.5°. Very sol. boiling EtOH. Sol.  $Et_2O$ , AcOEt. Spar. sol. cold  $H_2O$ . Volatile in steam. Conc.  $H_2SO_4 + HCl \rightarrow$  yellow sol.

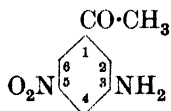
*Semicarbazone*: golden-yellow prisms from AcOH. M.p. 223°.

Bamberger, *Ber.*, 1915, 48, 562.

**5-Nitro-*o*-aminoacetophenone.**

Yellow needles from  $\text{H}_2\text{O}$ . M.p. 150–1. Very sol. boiling EtOH. Mod. sol.  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ . Non-volatile in steam.

See previous reference.

**5-Nitro-*m*-aminoacetophenone**

$\text{C}_8\text{H}_8\text{O}_3\text{N}_2$

MW, 180

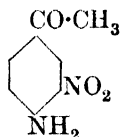
Yellow needles from  $\text{H}_2\text{O}$ . M.p. 156–8°.

Berend, Heymann, *J. prakt. Chem.*, 1904, 69, 471.

**6-Nitro-*m*-aminoacetophenone.**

*N*-Di-Me:  $\text{C}_{10}\text{H}_{12}\text{O}_3\text{N}_2$ . MW, 208. Yellow needles with steel-blue reflex from EtOH.Aq. M.p. 149–50°. Mod. sol. EtOH. Less sol.  $\text{H}_2\text{O}$ .

Rupe, Braun, v. Zembrzuski, *Ber.*, 1901, 34, 3525.

**3-Nitro-*p*-aminoacetophenone**

$\text{C}_8\text{H}_8\text{O}_3\text{N}_2$

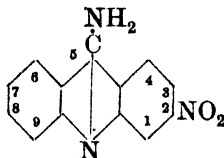
MW, 180

Yellow needles from EtOH.Aq., reddish-brown cryst. from toluene. M.p. 153–4° (148–9°).

*N*-Acetyl: yellow needles from EtOH. M.p. 140–1° (137°).

Mayer, Stark, Schön, *Ber.*, 1932, 65, 1335.

Gibson, Levin, *J. Chem. Soc.*, 1931, 2403.

**2-Nitro-5-aminoacridine**

$\text{C}_{13}\text{H}_9\text{O}_2\text{N}_3$

MW, 239

Cryst. M.p. above 300° decomp.

M.L.B., D.R.P., 364,037, (*Chem. Zentr.*, 1923, II, 1251).

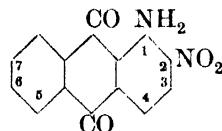
I.G., D.R.P., 304,280, (*Chem. Zentr.*, 1930, II, 626).

**6-Nitro-3-aminoanisic Acid.**

See under 6-Nitro-4-hydroxy-*m*-aminobenzoic Acid.

**Nitroaminoanisole.**

See Nitroanisidine.

**2-Nitro-1-aminoanthraquinone**

$\text{C}_{14}\text{H}_8\text{O}_4\text{N}_2$

MW, 268

Orange-red cryst. Sol. Py. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.  $\rightarrow$  violet-red on adding boric acid.

*Urethane*: cryst. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

Drescher, Thomas, U.S.P., 1,528,470, (*Chem. Abstracts*, 1926, 20, 425).

Bayer, D.R.P., 167,410, (*Chem. Zentr.*, 1906, I, 1065).

**3-Nitro-1-aminoanthraquinone.**

Dark red needles from EtOH. M.p. 265°.

Dhar, *J. Chem. Soc.*, 1920, 117, 1003.

**4-Nitro-1-aminoanthraquinone.**

Yellowish-red needles from  $\text{PhNO}_2$ . M.p. 296°. Spar. sol. EtOH. Insol.  $\text{H}_2\text{O}$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*N*-Me:  $\text{C}_{15}\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 282. Reddish-brown cryst. M.p. 250°. Sol.  $\text{Me}_2\text{CO}$ , Py. Spar. sol. EtOH, AcOH. *N*-Acetyl: orange prisms. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*N*-Acetyl: golden-yellow cryst. from AcOH or Py. M.p. 256–8°. Sol. AcOH, Py, aniline. Spar. sol. EtOH,  $\text{Et}_2\text{O}$ , ligroin. Insol.  $\text{H}_2\text{O}$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*Urethane*: cryst. from AcOH.

Bayer, D.R.P., 125,391, (*Chem. Zentr.*, 1901, II, 1219).

Noelting, Wortmann, *Ber.*, 1906, 39, 643.

**5-Nitro-1-aminoanthraquinone.**

Red cryst. from EtOH. M.p. 293° decomp. Sol. AcOH, boiling toluene. Insol.  $\text{Et}_2\text{O}$ , ligroin.

*N*-Me:  $\text{C}_{15}\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 282. Violet-black needles from AcOH. M.p. 250–2°. Very sol.  $\text{PhNO}_2$ . Sol. AcOH. Spar. sol.  $\text{C}_6\text{H}_6$ , EtOH.

*N*-Acetyl: yellow cryst. from chlorobenzene. M.p. 275°. Very sol.  $\text{PhNO}_2$ . Sol. hot  $\text{CHCl}_3$ . Spar. sol.  $\text{C}_6\text{H}_6$ , EtOH. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*N*-Et:  $\text{C}_{16}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 296. Black needles from AcOH. M.p. 238°. Very sol.  $\text{PhNO}_2$ . Sol. chlorobenzene. Spar. sol. EtOH, AcOH. *N*-Acetyl: orange-yellow plates from chlorobenzene. M.p. 242°. Very sol.  $\text{PhNO}_2$ . Sol. chlorobenzene. Spar. sol. EtOH, AcOH.

N-p-Tolyl:  $C_{21}H_{14}O_4N_2$ . MW, 358. Brownish-violet plates from AcOH. Cold conc.  $H_2SO_4 \rightarrow$  yellowish-brown sol.

N- $\alpha$ -Naphthyl:  $C_{24}H_{16}O_4N_2$ . MW, 396. Brownish-violet plates from AcOH. Cold conc.  $H_2SO_4 \rightarrow$  green col.

N-Benzoyl: brown cryst. from o-dichlorobenzene. M.p. 236.5–237°.

N-o-Chlorobenzoyl: yellowish-brown prisms. M.p. 265–6°.

N-m-Methoxybenzoyl: yellowish prisms. M.p. 199–200°.

N-Anisoyl: yellow needles. M.p. 255–6°.

Ullmann, Kertész, *Ber.*, 1919, **52**, 556.

M.L.B., D.R.P., 292,395, (*Chem. Zentr.*, 1916, II, 41).

Hefti, *Helv. Chim. Acta*, 1931, **14**, 1409.

### 7-Nitro-1-aminoanthraquinone.

N-p-Tolyl:  $C_{21}H_{14}O_4N_2$ . MW, 358. Violet-black needles from  $Me_2CO$ . Cold conc.  $H_2SO_4 \rightarrow$  brownish-yellow sol.  $\rightarrow$  reddish-brown on heating.

Bayer, D.R.P., 126,542, (*Chem. Zentr.*, 1901, II, 1373).

### 8-Nitro-1-aminoanthraquinone.

Red cryst. Fuming sulphuric acid (40%  $SO_3$ )  $\rightarrow$  yellow sol.

N-Me:  $C_{15}H_{10}O_4N_2$ . MW, 282. Red needles from Py.

N-Di-Me:  $C_{16}H_{12}O_4N_2$ . MW, 296. Cryst. from Py.  $CHCl_3 \rightarrow$  bluish-red sol. Conc.  $H_2SO_4 \rightarrow$  colourless sol.

N-Et:  $C_{16}H_{12}O_4N_2$ . MW, 296. Brown needles with green reflex.

N-Di-Et:  $C_{18}H_{16}O_4N_2$ . MW, 324. Needles.  $CHCl_3 \rightarrow$  bluish-red sol. Conc.  $H_2SO_4 \rightarrow$  yellow sol.  $\rightarrow$  violet on heating with boric acid.

N-Phenyl: 8-nitro-1-anilinoanthraquinone.  $C_{20}H_{12}O_4N_2$ . MW, 344. Bluish-violet needles from AcOH.

N-p-Tolyl:  $C_{21}H_{14}O_4N_2$ . MW, 358. Cryst. from AcOH. Sol.  $Me_2CO$ . Conc.  $H_2SO_4 \rightarrow$  brown sol.  $\rightarrow$  violet-blue on heating.

N-Benzoyl: brownish-green needles from o-dichlorobenzene. M.p. 266.5–267.5°.

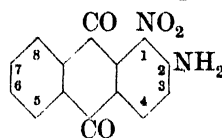
N-o-Chlorobenzoyl: brownish-green prisms. M.p. 253–4°.

N-Anisoyl: brown prisms. M.p. 246.5–247.5°.

Bayer, D.R.P., 144,634, (*Chem. Zentr.*, 1903, II, 750); D.R.P., 147,851, (*Chem. Zentr.*, 1904, I, 132).

Hefti, *Helv. Chim. Acta*, 1931, **14**, 1410.

### 1-Nitro-2-aminoanthraquinone



$C_{14}H_8O_4N_2$

MW, 268

Light green plates from AcOH or  $PhNO_2$ . M.p. 310°. Sol. Py, quinoline. Mod. sol.  $Me_2CO$ . Spar. sol. EtOH,  $Et_2O$ . Sol. 20 parts boiling  $PhNO_2$ . Conc.  $H_2SO_4 \rightarrow$  yellow sol.

N-Acetyl: needles from AcOH. M.p. 277–8°. Sol.  $PhNO_2$ . Spar. sol. EtOH, AcOH. Insol.  $Et_2O$ . Conc.  $H_2SO_4 \rightarrow$  green sol.

Urethane: cryst. from  $Me_2CO$ . M.p. 205°. Sol. boiling AcOH, toluene. Spar. sol. EtOH. Almost insol.  $Et_2O$ .

Ullmann, Medenwald, *Ber.*, 1913, **46**, 1806.

Chem. Fabr. Griesheim-Elektron, D.R.P., 259,432, (*Chem. Zentr.*, 1913, I, 1742).

### 3-Nitro-2-aminoanthraquinone.

Yellowish-brown cryst. from  $PhNO_2$ . M.p. 316–17° (305–6°). Sol.  $PhNO_2$ . Spar. sol. EtOH,  $C_6H_6$ , Py, xylene. Conc.  $H_2SO_4 \rightarrow$  orange sol.

Urethane: cryst. from toluene. M.p. 225°. Sol. boiling AcOH, toluene. Spar. sol. EtOH. Insol.  $Et_2O$ .

See first reference above and also

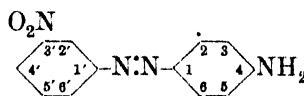
Badische, D.R.P., 148,109, (*Chem. Zentr.*, 1904, I, 230).

### 5-Nitro-2-aminoanthraquinone.

Orange-red cryst. from  $PhNO_2$ . M.p. 274°. Mod. sol.  $PhNO_2$ , chlorobenzene. Less sol. AcOH.

Eckert, *Monatsh.*, 1914, **35**, 296.

### 3'-Nitro-4-aminoazobenzene



$C_{12}H_{10}O_2N_4$

MW, 242

Orange leaflets from EtOH.Aq. M.p. 212–13°. Sol. EtOH,  $Me_2CO$ ,  $C_6H_6$ .

$B_2H_2PtCl_6$ : brick-red ppt. M.p. about 210° decomp.

Meldola, *J. Chem. Soc.*, 1884, **45**, 112.

### 4'-Nitro-4-aminoazobenzene.

Cryst. from toluene. M.p. 216° (203–5°). Spar. sol. cold EtOH.

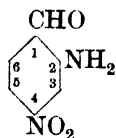
N-Me:  $C_{13}H_{12}O_2N_4$ . MW, 256. Blue prisms

and needles from  $C_6H_6$ , plates from EtOH or AcOH. M.p. 206–7°. Boiling dil. HCl  $\rightarrow$  red sol. Dichroic. N-Acetyl: orange-red needles. M.p. 194–5°.

N-Acetyl: orange-red needles. M.p. 245–6°.

Witt, Kopetschni, *Ber.*, 1912, **45**, 1148.

#### 4-Nitro-*o*-aminobenzaldehyde



$C_7H_6O_3N_2$  MW, 166

Needles from hot  $H_2O$ . M.p. 124°. Slowly reduces  $NH_3 \cdot AgNO_3$ .

Oxime: light yellow cryst. from  $Me_2CO \cdot Aq$ . M.p. 193°.

Semicarbazone: light yellow cryst. from AcOH. Decomp. at 390°.

Anil: red cryst. from hot EtOH. M.p. 147°.

Sachs, Sichel, *Ber.*, 1904, **37**, 1862.

#### 5-Nitro-*o*-aminobenzaldehyde.

Yellow prisms from EtOH. M.p. 200–5°. Sol. hot  $H_2O$ .

N-Di-Me:  $C_9H_{10}O_3N_2$ . MW, 194. Yellow needles from ligroin. M.p. 105°. Oxime: m.p. 125°.

Oxime: yellow cryst. from EtOH. M.p. 203°.

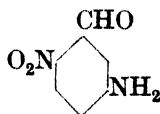
N-Acetyl: needles from  $H_2O$ . M.p. 160–1°. Sol.  $H_2O$ , EtOH. Oxime: needles from EtOH. M.p. 239°.

N-p-Toluenesulphonyl: m.p. 181–2°.

Cohn, Springer, *Monatsh.*, 1903, **24**, 98.

General Aniline Works, U.S.P., 1,876,955, (*Chem. Abstracts*, 1933, **27**, 993).

#### 6-Nitro-*m*-aminobenzaldehyde



$C_7H_6O_3N_2$  MW, 166

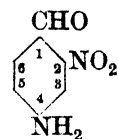
Yellow needles from  $H_2O$ .

N-Acetyl: yellow needles from  $H_2O$ , EtOH. Aq., or xylene. M.p. 161°. Oxime: reddish-yellow needles. M.p. 189°. Sol. EtOH. Phenylhydrazones: red cryst. from  $Me_2CO$ . M.p. 247°. Sol. EtOH. Spar. sol.  $C_6H_6$ . Insol.  $H_2O$ .

Phenylhydrazones: red needles from EtOH. Aq. M.p. 212°.

Friedländer, Fritsch, *Monatsh.*, 1903, **24**, 8.

#### 2-Nitro-*p*-aminobenzaldehyde



$C_7H_6O_3N_2$  MW, 166

Oxime: orange-yellow needles from 50% EtOH. M.p. 177–8°.

Semicarbazone: red cryst. from  $H_2O$ . Decomp. at 330°.

Phenylhydrazones: light red needles from EtOH. M.p. 163°.

Sachs, Kempf, *Ber.*, 1902, **35**, 1234, 2705.

#### 3-Nitro-*p*-aminobenzaldehyde.

Yellow needles from  $H_2O$ . M.p. 190–5–191°. Sol. EtOH. Spar. sol.  $H_2O$ . Insol.  $Et_2O$ .

N-Di-Me:  $C_9H_{10}O_3N_2$ . MW, 194. Yellow needles from EtOH. M.p. 105°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Mod. sol. hot ligroin. Conc.  $H_2SO_4 \rightarrow$  yellow sol. Oxime: orange prisms. M.p. 132°.

N-Acetyl: yellow needles from  $H_2O$ . M.p. 155°. Sol. warm EtOH, hot  $H_2O$ . Spar. sol.  $Et_2O$ . Oxime: yellow needles from EtOH. M.p. 206°. Phenylhydrazones: red cryst. from AcOH. M.p. 209°. p-Nitrophenylhydrazones: orange needles from EtOH. M.p. 289–90°. Phenylhydrazones: reddish-brown plates. M.p. 202°.

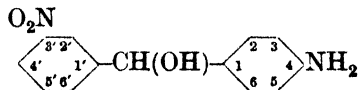
Oxime: orange needles from EtOH. Aq. M.p. 207°. Dil. NaOH  $\rightarrow$  deep red sol.

p-Nitrophenylhydrazones: dark maroon needles from AcOH. M.p. 270–2°.

Cohn, Springer, *Monatsh.*, 1903, **24**, 92.

Hodgson, Beard, *J. Chem. Soc.*, 1927, 23

#### 3'-Nitro-4-aminobenzhydrol



$C_{13}H_{12}O_3N_2$  MW, 244

N-Di-Me:  $C_{15}H_{16}O_3N_2$ . MW, 272. Yellow cryst. M.p. 74°. Sol. EtOH. Insol.  $H_2O$ .

N-Di-Et:  $C_{17}H_{20}O_3N_2$ . MW, 300. Yellow cryst. M.p. 65°. Sol. EtOH. Insol.  $H_2O$ .

Kalle, D.R.P., 45,806.

#### 4'-Nitro-4-aminobenzhydrol.

Only known as polymer. M.p. about 240°. Sol. AcOH-HCl. Insol. EtOH,  $Et_2O$ ,  $C_6H_6$ , AcOH.

N-Me:  $C_{14}H_{14}O_3N_2$ . MW, 258. Yellow

needles from 25% EtOH. M.p. 113° (108°). Sol. EtOH.

*N-Di-Me*:  $C_{15}H_{16}O_3N_2$ . MW, 272. Yellow needles from EtOH.Aq. M.p. 96°. Sol. most solvents. Insol.  $H_2O$ , ligroin. *Methiodide*: cryst. M.p. 175° decomp.

*N-Et*:  $C_{15}H_{16}O_3N_2$ . MW, 272. Yellow cryst. M.p. 99°. Sol. EtOH. Insol.  $H_2O$ .

*N-Di-Et*:  $C_{17}H_{20}O_3N_2$ . MW, 300. Yellow cryst. M.p. 92°. Sol. EtOH. Insol.  $H_2O$ .

Esselen, Clarke, *J. Am. Chem. Soc.*, 1914, **36**, 314.

Albrecht, *Ber.*, 1888, **21**, 3294.

Kalle, D.R.P., 119,461, (*Chem. Zentr.*, 1901, I, 866).

See also previous reference.

### Nitro-*o*-aminobenzoic Acid.

See Nitroanthranilic Acid.

### 2-Nitro-*m*-aminobenzoic Acid



$C_7H_6O_4N_2$  MW, 182

Golden-yellow needles from  $H_2O$  or EtOH.Aq. M.p. 156–7°. Sol. hot  $H_2O$ , EtOH,  $Et_2O$ , AcOH. Insol. ligroin.

*N-Acetyl*: needles or plates. M.p. 240–1° decomp. Sol. EtOH,  $Me_2CO$ , AcOH.

Kaiser, *Ber.*, 1885, **18**, 2950.

### 4-Nitro-*m*-aminobenzoic Acid.

Red plates or needles from EtOH. M.p. 298° decomp. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

*Et ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Red needles from EtOH or AcOH. M.p. 139°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ , AcOH.

*Amide*:  $C_7H_7O_3N_3$ . MW, 181. Reddish-yellow needles from boiling  $H_2O$ . M.p. 231–2°. Sol. hot  $H_2O$ , EtOH, AcOH.

*N-Me*:  $C_8H_8O_4N_2$ . MW, 196. Red leaflets from EtOH. M.p. 268° decomp. *Methylamide*:  $C_9H_{11}O_3N_3$ . MW, 209. Needles from hot  $H_2O$ . M.p. 194°. Sol. hot  $H_2O$ , EtOH, AcOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ .

*N-Acetyl*: yellow plates. M.p. 205–6°. Sol. boiling EtOH,  $Me_2CO$ , AcOH. Spar. sol. cold  $H_2O$ .

Thieme, *J. prakt. Chem.*, 1891, **43**, 464.

See also previous reference.

### 5-Nitro-*m*-aminobenzoic Acid.

Golden-yellow prisms from  $H_2O$ . M.p. 209–10° (208°). Very sol. hot AcOH. Sol. EtOH. Less sol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ .  $k = 2.1 \times 10^{-4}$  at 25°.

*Me ester*:  $C_8H_8O_4N_2$ . MW, 196. Yellow needles from  $H_2O$ , prisms from EtOH. M.p. 160°. Very sol. EtOH,  $Et_2O$ . Less sol.  $C_6H_6$ . *N-Acetyl*: cryst. M.p. 165–7°. *N-Benzoyl*: m.p. 178°.

*Et ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Yellow needles from EtOH. M.p. 155°. Sol. EtOH. Spar. sol.  $H_2O$ . *N-Acetyl*: cryst. from EtOH.Aq. M.p. 168°. Very sol. pet. ether.

*Hydrazide*: reddish-yellow plates from  $H_2O$ . M.p. 221°. Sol. AcOH. Spar. sol. hot EtOH. Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , ligroin. Reduces warm Fehling's and  $NH_3 \cdot AgNO_3$ .

*N-Et*:  $C_9H_{10}O_4N_2$ . MW, 210. Yellow needles from  $H_2O$ . M.p. 208°. Almost insol.  $H_2O$ .

Hübner, *Ann.*, 1884, **222**, 81.

Curtius, Riedel, *J. prakt. Chem.*, 1907, **76**, 255.

Cohen, McCandlish, *J. Chem. Soc.*, 1905, **87**, 1267.

### 6-Nitro-*m*-aminobenzoic Acid.

Yellow needles or prisms from  $H_2O$ . M.p. 235° decomp. Sol. EtOH. Spar. sol. hot  $H_2O$ ,  $Et_2O$ .

*Et ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Yellow cryst. from EtOH.Aq. M.p. 107–5°.

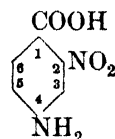
*N-Acetyl*: brownish-red cryst. from  $H_2O$ , needles from AcOH. M.p. 225°. Sol. most org. solvents.

*Hydrazide*: yellow prisms from EtOH. Sol.  $H_2O$ , AcOH. Mod. sol. EtOH. Almost insol.  $Et_2O$ .

Hewitt, Mitchell, *J. Chem. Soc.*, 1907, **91**, 1258.

Kalb, Gross, *Ber.*, 1926, **59**, 736.

### 2-Nitro-*p*-aminobenzoic Acid



$C_7H_6O_4N_2$  MW, 182

Red needles from hot  $H_2O$ . M.p. 255° (234–5°). Sol. EtOH, AcOH. Spar. sol. cold  $H_2O$ . Sweet taste.

*Me ester*:  $C_8H_8O_4N_2$ . MW, 196. Light brown prisms from EtOH.Aq. M.p. 157–159.5°. *N-Acetyl*: needles from  $H_2O$ . M.p. 76°. *N-Benzoyl*: needles from  $C_6H_6$ . M.p. 93–4°.

*Et ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Pale yellow needles from hot  $H_2O$ . M.p. 130°. Sol. EtOH,  $Et_2O$ .

*Anilide*: needles from hot  $H_2O$ . M.p. 226°.

Spar. sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . *N*-Acetyl: yellow needles from  $\text{H}_2\text{O}$ . M.p. 238°.

*Hydrazide*: golden-yellow plates from hot  $\text{H}_2\text{O}$ . M.p. 212°. Sol. EtOH. Spar. sol. AcOH. Insol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ ,  $\text{Et}_2\text{O}$ , ligroin. Reduces warm Fehling's and  $\text{NH}_3\cdot\text{AgNO}_3$ .

*N*-Acetyl: light yellow needles from EtOH. M.p. 219°.

Bogert, Kropff, *J. Am. Chem. Soc.*, 1909, **31**, 847.

Curtius, Bollenbach, *J. prakt. Chem.*, 1907, **76**, 288.

Cohen, McCandlish, *J. Chem. Soc.*, 1905, **87**, 1268.

Höchst, D.R.P., 204,884, (*Chem. Zentr.*, 1909, I, 474).

### 3-Nitro-*p*-aminobenzoic Acid.

Reddish-yellow needles from EtOH. M.p. 284°. Spar. sol. hot EtOH.

*Me ester*:  $\text{C}_8\text{H}_8\text{O}_4\text{N}_2$ . MW, 196. Yellow cryst. from  $\text{Me}_2\text{CO}\cdot\text{Aq}$ . M.p. 199.5–200°.

*Et ester*:  $\text{C}_9\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 210. Yellow needles from EtOH. M.p. 145°. Sol. usual org. solvents. *N*-Acetyl: m.p. 96–7°. *N*-Chloroacetyl: yellow needles from EtOH or  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 102°.

*Amide*:  $\text{C}_7\text{H}_7\text{O}_3\text{N}_3$ . MW, 181. Yellow needles from EtOH. M.p. 227°. Sol.  $\text{Me}_2\text{CO}$ . Spar. sol. hot EtOH. Insol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , pet. ether. *N*-Acetyl: yellow needles from EtOH. M.p. 239.5° (sealed tube). Sol. boiling  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Sublimes.

*Nitrile*:  $\text{C}_7\text{H}_5\text{O}_2\text{N}_3$ . MW, 163. Yellow needles. M.p. 159–60°. Sol. hot  $\text{H}_2\text{O}$ , EtOH. Less sol.  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . *N*-Acetyl: light yellow needles from EtOH. M.p. 131.5°. Sol.  $\text{H}_2\text{O}$ , EtOH. Less sol.  $\text{C}_6\text{H}_6$ . Insol.  $\text{CCl}_4$ .

*Anilide*: red needles from EtOH. M.p. 215–16°. Sol. EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , AcOH. Insol. pet. ether.

*N*-Me: see 3-Nitro-*p*-methylaminobenzoic Acid.

*N*-Di-Me: see 3-Nitro-*p*-dimethylaminobenzoic Acid.

*N*-Et:  $\text{C}_9\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 210. Golden-yellow needles from  $\text{H}_2\text{O}$ , EtOH or ligroin. M.p. 239–40°. *Et ester*:  $\text{C}_{11}\text{H}_{14}\text{O}_4\text{N}_2$ . MW, 238. Yellow needles. M.p. 92°. Sol. EtOH, AcOH. Insol.  $\text{H}_2\text{O}$ .

*N*-Di-Et:  $\text{C}_{11}\text{H}_{14}\text{O}_4\text{N}_2$ . MW, 238. Reddish-yellow needles from ligroin. M.p. 117°.

*N*-Phenyl: see 2-Nitrodiphenylamine-4-carboxylic acid.

*N*-o-Tolyl: see 2-Nitro-2'-methyldiphenylamine-4-carboxylic acid.

*N*-p-Tolyl: see 2-Nitro-4'-methyldiphenylamine-4-carboxylic acid.

*N*- $\alpha$ -Naphthyl:  $\text{C}_{17}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 308. Reddish-brown powder. Sol. usual solvents. *Et ester*:  $\text{C}_{19}\text{H}_{16}\text{O}_4\text{N}_2$ . MW, 336. Reddish-brown plates from EtOH.Aq. M.p. 109°. Sol. EtOH,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , AcOH.

*N*- $\beta$ -Naphthyl:  $\text{C}_{17}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 308. Dark red powder. Sol. EtOH,  $\text{Me}_2\text{CO}$ , AcOH. Less sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . *Et ester*:  $\text{C}_{19}\text{H}_{16}\text{O}_4\text{N}_2$ . MW, 336. Light yellow needles from EtOH. M.p. 127.5°. Sol. EtOH,  $\text{Me}_2\text{CO}$ , AcOH,  $\text{CHCl}_3$ .

*N*-Formyl: needles from EtOH. M.p. 221° decomp. Mod. sol. EtOH. Insol.  $\text{H}_2\text{O}$ .

*N*-Acetyl: yellow plates. M.p. 220–1°. Sol. boiling EtOH,  $\text{Me}_2\text{CO}$ , AcOH. Spar. sol. cold  $\text{H}_2\text{O}$ .

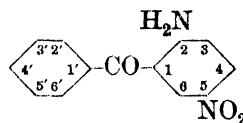
Borsche, Stackmann, Makaroff-Semljanski, *Ber.*, 1916, **49**, 2232.

Bogert, Wise, *J. Am. Chem. Soc.*, 1910, **32**, 1497.

Thieme, *J. prakt. Chem.*, 1891, **43**, 456.

Reverdin, de Luc, *Ber.*, 1909, **42**, 1726.

### 5-Nitro-2-aminobenzophenone



$\text{C}_{13}\text{H}_{10}\text{O}_3\text{N}_2$

MW, 242

Yellowish-red prisms with blue reflex. M.p. 161.5°. Sol. EtOH, AcOH.

*N*-Phenyl:  $\text{C}_{19}\text{H}_{14}\text{O}_3\text{N}_2$ . MW, 318. Yellow leaflets from EtOH. M.p. 155°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin.

*N*-o-Methoxyphenyl:  $\text{C}_{20}\text{H}_{16}\text{O}_4\text{N}_2$ . MW, 348. Pale yellow needles or greenish-yellow leaflets from EtOH. M.p. 139°.

Ullmann, Mallet, *Ber.*, 1898, **31**, 1695.

Ullmann, Ernst, *Ber.*, 1906, **39**, 300.

### 2'-Nitro-2-aminobenzophenone.

Brownish-yellow needles from EtOH. M.p. 149–50°.

Heyl, *Ber.*, 1898, **31**, 3033.

### 5-Nitro-3-aminobenzophenone.

Exists in two forms.

(i) Orange needles from EtOH.Aq., plates from  $\text{C}_6\text{H}_6$ . M.p. 130°.

(ii) Cryst. from EtOH. M.p. 146°.

*Diacyl*: pale yellow needles from EtOH.Aq. M.p. 191°.

Waters, *J. Chem. Soc.*, 1929, 2110.



**3-Nitro-4-aminobenzophenone.**

Yellow needles from EtOH. M.p. 140-5° (135°). Sol. AcOH, CHCl<sub>3</sub>, Py. Spar. sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, Et<sub>2</sub>O, CS<sub>2</sub>.

N-Di-Me: C<sub>15</sub>H<sub>14</sub>O<sub>3</sub>N<sub>2</sub>. MW, 270. Golden-yellow cryst. from EtOH. M.p. 116°. Spar. sol. ligroin.

N-Et: C<sub>15</sub>H<sub>14</sub>O<sub>3</sub>N<sub>2</sub>. MW, 270. Yellow needles. M.p. 99-100°.

N-Phenyl: C<sub>19</sub>H<sub>14</sub>O<sub>3</sub>N<sub>2</sub>. MW, 318. Orange needles. M.p. 157°.

N-Acetyl: pale yellow needles from EtOH. Spar. sol. EtOH, Et<sub>2</sub>O. Mod. sol. C<sub>6</sub>H<sub>6</sub>.

N-Benzoyl: plates from Me<sub>2</sub>CO. M.p. 154-5°. Sol. C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Mod. sol. Me<sub>2</sub>CO. Spar. sol. EtOH, ligroin. Insol. H<sub>2</sub>O.

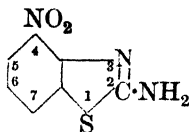
Schöpf, *Ber.*, 1891, 24, 3772.

Maron, Fox, *Ber.*, 1914, 47, 2778.

**3'-Nitro-4-aminobenzophenone.**

N-Di-Me: C<sub>15</sub>H<sub>14</sub>O<sub>3</sub>N<sub>2</sub>. MW, 270. Yellow cryst. M.p. 173°. Spar. sol. EtOH.

Höchst, D.R.P., 42,853.

**4-Nitro-2-aminobenzthiazole (3-Nitro-1-aminobenzthiazole)**

C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>N<sub>3</sub>S MW, 195

Yellow plates from EtOH-AcOEt. M.p. 233° decomp.

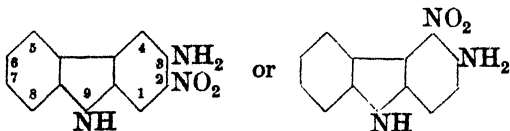
Dyson, Hunter, Morris, *J. Chem. Soc.*, 1927, 1192.

**6-Nitro-2-aminobenzthiazole (5-Nitro-1-aminobenzthiazole).**

Orange, microcryst. powder from AcOEt. M.p. 243°.

Acetyl: pale yellow cryst. powder. M.p. 292°.

Hunter, Jones, *J. Chem. Soc.*, 1930, 2203.

**2-(or 4)-Nitro-3-aminocarbazole**

C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>N<sub>3</sub> MW, 227

Dark violet needles from EtOH. M.p. 233°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, AcOH.

3-N-Acetyl: red needles from EtOH. M.p. 274°. Sol. hot EtOH. Insol. H<sub>2</sub>O.

Diacetyl: greenish-yellow cryst. from AcOH. M.p. 226°. Sol. hot AcOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

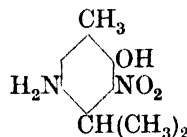
Kehrmann, Zweifel, *Helv. Chim. Acta*, 1928, 11, 1213.

**4-(or 2)-Nitro-3-aminocarbazole.**

Dark reddish-brown needles from EtOH. M.p. 177°. Sol. hot EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

3-N-Acetyl: reddish-brown needles. M.p. 198°. Sol. EtOH, AcOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

See previous reference.

**3-Nitro-5-aminocarbavacrol**

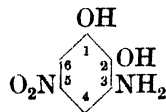
C<sub>10</sub>H<sub>14</sub>O<sub>3</sub>N<sub>2</sub> MW, 210

Yellow cryst from EtOH. M.p. 134-5°.

O-Benzoyl: cryst. M.p. about 280-3°.

O:N-Diacetyl: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 222-5°.

Soderi, *Gazz. chim. ital.*, 1895, 25, ii, 406.

**5-Nitro-3-aminocatechol**

C<sub>6</sub>H<sub>6</sub>O<sub>4</sub>N<sub>2</sub> MW, 170

1-Me ether: 5-nitro-3-aminoguaiacol. C<sub>7</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub>. MW, 184. Brown needles from H<sub>2</sub>O. M.p. 182° decomp. N-Acetyl: needles + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 224-6° decomp. O:N-Diacetyl: needles from H<sub>2</sub>O. M.p. 204° decomp.

1:2-Di-Me ether: 5-nitro-3-aminoveratrol. C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 198. Brown needles from MeOH.Aq. M.p. 105-6°. Sol. most org. solvents. Spar. sol. cold H<sub>2</sub>O. N-Acetyl: yellowish plates from EtOH. M.p. 172-3°. N-Benzoyl: needles from AcOH. M.p. 145-6°.

Meldola, Woolcott, Wray, *J. Chem. Soc.*, 1896, 69, 1331.

Gibson, Simonsen, Rau, *J. Chem. Soc.*, 1917, 111, 75.

**3-Nitro-4-aminocatechol.**

2-Me ether: 6-nitro-5-aminoguaiacol. C<sub>7</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub>. MW, 184. Light red needles from C<sub>6</sub>H<sub>6</sub> or H<sub>2</sub>O. M.p. 169-71°. N-Acetyl: orange-red cryst. from EtOH. M.p. 223°. O:N-Diacetyl: yellow plates or needles from H<sub>2</sub>O or EtOH. M.p. 158°. O:N-Dibenzoyl: yellow needles from EtOH. M.p. 177°.

1:2-Di-Me ether: 3-nitro-4-aminoveratrol.

$C_8H_{10}O_4N_2$ . MW, 198. Red needles from EtOH.Aq. M.p. 74°. Spar. sol. pet. ether. Dichroic.

Fichter, Schwab, *Ber.*, 1906, **39**, 3340.

Pisovschi, *Ber.*, 1910, **43**, 2142.

#### 5-Nitro-4-aminocatechol.

1:2-*Di-Me ether*: 5-nitro-4-aminoveratrol.  $C_8H_{10}O_4N_2$ . MW, 198. Orange prisms or needles from EtOH. M.p. 175° (171°). Sol. hot EtOH,  $Me_2CO$ , hot AcOEt. Spar. sol.  $C_6H_6$ ,  $CHCl_3$ . Sol. conc. min. acids. *N-Acetyl*: yellow needles from EtOH. M.p. 199° (196°). Spar. sol. EtOH, AcOH. Insol.  $H_2O$ . *N-Benzoyl*: yellow needles from EtOH. M.p. 153-4°.

Simonsen, Rau, *J. Chem. Soc.*, 1918, **113**, 27.

Jones, Robinson, *J. Chem. Soc.*, 1917, **111**, 914.

#### 6-Nitro-4-aminocatechol.

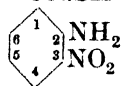
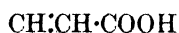
Yellowish-orange needles from  $H_2O$ . M.p. 228°. Sol. EtOH.

O:O:*N-Triacetyl*: needles from AcOH. M.p. 207°. Sol. EtOH, AcOEt. Spar. sol.  $C_6H_6$ .

Meldola, Woolcott, Wray, *J. Chem. Soc.*, 1896, **69**, 1334.

Heller, Lindner, Georgi, *Ber.*, 1923, **56**, 1872.

#### 3-Nitro-*o*-aminocinnamic Acid



$C_9H_8O_4N_2$  MW, 208

*Amide*:  $C_9H_9O_3N_3$ . MW, 207. Cryst. in plates. Heat with HCl in sealed tube to 140° → 8-nitrocarbostyryl.

v. Miller, Kinkeln, *Ber.*, 1889, **22**, 1711.

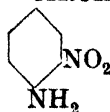
#### 4-Nitro-*o*-aminocinnamic Acid.

Light reddish-brown needles. M.p. 240°. Sol. EtOH,  $Me_2CO$ . Spar. sol.  $H_2O$ . Insol.  $Et_2O$ ,  $C_6H_6$ , ligroin. With HCl in sealed tube at 150° → 7-nitrocarbostyryl.

*Et ester*:  $C_{11}H_{12}O_4N_2$ . MW, 236. Dark reddish-brown needles. M.p. 158-60°.

Friedländer, Lazarus, *Ann.*, 1885, **229**, 242.

#### 3-Nitro-*p*-aminocinnamic Acid



$C_9H_8O_4N_2$

MW, 208

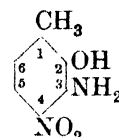
Red needles from  $H_2O$ . M.p. 224.5° (218°). Sol. EtOH, AcOH. Mod. sol.  $H_2O$ . Prac. insol.  $C_6H_6$ , ligroin.

*N-Acetyl*: yellow cryst. M.p. 261-6°.

Gabriel, Herzberg, *Ber.*, 1883, **16**, 2042.

Cohn, Springer, *Monatsh.*, 1903, **24**, 94.

#### 4-Nitro-3-amino-*o*-cresol



$C_7H_8O_3N_2$  MW, 168

*Me ether*:  $C_8H_{10}O_3N_2$ . MW, 182. Needles from MeOH. M.p. 72°. Sol. most org. solvents. Volatile in steam. *N-Acetyl*: prisms from  $H_2O$ . M.p. 170-1°. Sol.  $H_2O$ .

Simonsen, Nayak, *J. Chem. Soc.*, 1915, **107**, 831.

#### 5-Nitro-3-amino-*o*-cresol.

Reddish-brown needles from  $C_6H_6$ . M.p. 165° decomp.

*Me ether*: yellow needles from MeOH. M.p. 113°. *N-Acetyl*: needles +  $H_2O$ . M.p. anhyd. 141-2°.

Cazeneuve, *Bull. soc. chim.*, 1897, **17**, 206.

Bovini, *Chem. Abstracts*, 1928, **22**, 1578.

See also previous reference.

#### 6-Nitro-3-amino-*o*-cresol.

*Me ether*: yellowish cryst. from MeOH. M.p. 103°. Sol. most org. solvents. *N-Acetyl*: needles from EtOH.Aq. M.p. 119-20°.

Simonsen, Nayak, *J. Chem. Soc.*, 1915, **107**, 830.

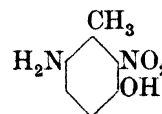
#### 3-Nitro-5-amino-*o*-cresol.

Reddish-brown needles from EtOH. M.p. 118°.

*N-Acetyl*: yellow needles from EtOH. M.p. 217°.

Nietzki, Ruppert, *Ber.*, 1890, **23**, 3477.

#### 2-Nitro-6-amino-*m*-cresol



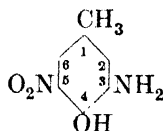
$C_7H_8O_3N_2$  MW, 168

Reddish-brown needles from EtOH. M.p. 201°. Sol. dil. acids and alkalis.

O: *N*-Diacetyl: needles. M.p. 127–8°.

Cohen, Marshall, *J. Chem. Soc.*, 1904, **85**, 527.

Brand, Zöller, *Ber.*, 1907, **40**, 3332.

**5-Nitro-3-amino-*p*-cresol**

$C_7H_8O_3N_2$  MW, 168

Reddish-brown cryst. from EtOH. M.p. 119° (110°).

*N*-Acetyl: yellow cryst. from EtOH. M.p. 143°.

Kehrmann, Winkelmann, *Ber.*, 1907, **40**, 618.

Lindemann, Romanoff, *J. prakt. Chem.*, 1929, **122**, 229.

**6-Nitro-3-amino-*p*-cresol.**

Yellow cryst. from EtOH. M.p. 199–200° decomp.

*Meth*: 6-nitrocresidine.  $C_8H_{10}O_3N_2$ . MW, 182. Yellow needles. M.p. 132°. *N*-Acetyl: needles from EtOH.Aq. M.p. 156°.

*N*-Acetyl: needles from AcOH. M.p. 242°.

Baranger, *Bull. soc. chim.*, 1931, **49**, 1215.

Limpach, *Ber.*, 1889, **22**, 790.

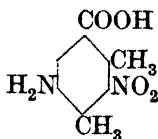
Bloxam, E.P., 168,681, (*Chem. Abstracts*, 1922, **16**, 500).

**Nitroamino-*N*-diethylaniline.**

See under Nitrophenylenediamine.

**Nitroamino-*N*-dimethylaniline.**

See under Nitrophenylenediamine.

**3-Nitro-5-amino-2:4-dimethylbenzoic Acid**

$C_9H_{10}O_4N_2$  MW, 210

Pale yellow prisms from EtOH. M.p. 251° decomp. Spar. sol.  $H_2O$ .

*B.HCl*: prisms. M.p. 250° decomp.

*Acetyl*: needles. M.p. 247°. Sol. hot EtOH. Mod. sol.  $H_2O$ .

Wheeler, Hoffmann, *Am. Chem. J.*, 1911, **45**, 440.

**Nitroamino-3:5-dimethylbenzoic Acid.**

See Nitroaminomesitylenic Acid.

**4-Nitro-6-amino-2:3-dimethyldiphenylamine.**

See under 5-Nitro-2:3-diamino-*p*-xylene.

**3-Nitro-2-aminodiphenyl**

$C_{12}H_{10}O_2N_2$

MW, 214

M.p. 44–5°.

*N*-Acetyl: m.p. 188°.

Sako, *Bull. Chem. Soc. Japan*, 1934, **9**, 55.

**5-Nitro-2-aminodiphenyl.**

Yellow needles from EtOH. M.p. 125°.

*N*-Acetyl: orange needles from EtOH. M.p. 133°.

*N*-*p*-Toluenesulphonyl: pale yellow needles from AcOH. M.p. 169°.

See previous reference and also

Bell, *J. Chem. Soc.*, 1928, 2774.

**2'-Nitro-2-aminodiphenyl.**

Free base not obtained pure.

*N*-Acetyl: cryst. from  $C_6H_6$ , m.p. 158°: cryst. from EtOH.Aq., m.p. 151–2°.

*N*-Diacetyl: yellow needles. M.p. 120–1° (125°).

*Picrate*: m.p. 167–5°.

Mascarelli, Gatti, Pirona, *Gazz. chim. ital.*, 1931, **61**, 786.

Mascarelli, Gatti, *Atti accad. Lincei*, 1929, **10**, 441; *Gazz. chim. ital.*, 1929, **59**, 861.

**4'-Nitro-2-aminodiphenyl.**

Orange-red needles from EtOH. M.p. 159°.

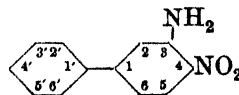
*N*-Acetyl: yellow needles from EtOH. M.p. 199°.

*N*-*p*-Toluenesulphonyl: pale yellow needles from AcOH. M.p. 163°.

Finzi, Bellavito, *Gazz. chim. ital.*, 1934, **64**, 343.

Scarborough, Waters, *J. Chem. Soc.*, 1927, 96.

Bell, *J. Chem. Soc.*, 1928, 2775.

**4-Nitro-3-aminodiphenyl**

$C_{12}H_{10}O_2N_2$

MW, 214

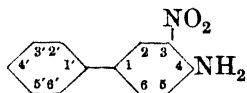
Orange needles from dil. EtOH. M.p. 116°.

*N*-Acetyl: yellow needles from EtOH. M.p. 115°.

Blakey, Scarborough, *J. Chem. Soc.*, 1927, 3008.

**3'-Nitro-3-aminodiphenyl.**

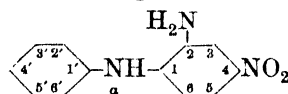
Not obtained pure. M.p. 115-40°.

*B.HCl*: m.p. 275-6° decomp.*N-Acetyl*: m.p. 156-7°.Mascarelli, Gatti, *Gazz. chim. ital.*, 1929, 59, 863; 1931, 61, 322.**4'-Nitro-3-aminodiphenyl.**Orange needles from EtOH. M.p. 137°.  
 $\text{CrO}_3 \rightarrow p\text{-nitrobenzoic acid.}$ *N-Acetyl*: yellow needles from EtOH. M.p. 192°.Blakey, Scarborough, *J. Chem. Soc.*, 1927, 3009.**3-Nitro-4-aminodiphenyl (3-Nitroxenylamine)** $\text{C}_{12}\text{H}_{10}\text{O}_2\text{N}_2$  MW, 214

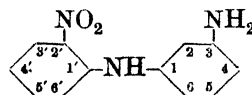
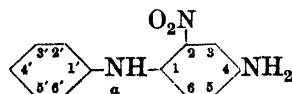
Red needles from EtOH. M.p. 167° (168-70°).

*N-Acetyl*: yellow needles from EtOH. M.p. 132°.*N-Benzoyl*: needles from AcOH. M.p. 143°.*N-Me*: red cryst. M.p. 112-13°.Fichter, Sulzberger, *Ber.*, 1904, 37, 881.Banús, Tomás, *Anales soc. españ. fis. quim.*, 1921, 19, 293.**2'-Nitro-4-aminodiphenyl (2'-Nitroxenylamine).**Red prisms from EtOH. M.p. 99°. Sol. EtOH. Prac. insol.  $\text{H}_2\text{O}$ .Finzi, Bellavito, *Gazz. chim. ital.*, 1934, 64, 342.Schultz, *Ann.*, 1874, 174, 225.Schultz, Schmidt, Strasser, *Ann.*, 1881, 207, 350.**3'-Nitro-4-aminodiphenyl (3'-Nitroxenylamine).**

Red prisms. M.p. 127°.

Finzi, Mangini, *Gazz. chim. ital.*, 1932, 62, 676.**4'-Nitro-4-aminodiphenyl (4'-Nitroxenylamine).**Red needles from EtOH. M.p. 200-1° (198°). Sol. hot EtOH. Prac. insol. hot  $\text{H}_2\text{O}$ .  $\text{CrO}_3 \rightarrow p\text{-nitrobenzoic acid.}$ *N-Acetyl*: yellow needles. M.p. 264° (240°).*N-Benzenesulphonyl*: yellow plates from dil. EtOH. M.p. 174°.Willstätter, Kalb, *Ber.*, 1906, 39, 3479.Schmidt, Schultz, *Ann.*, 1881, 207, 347.Codolosa, *Chem. Abstracts*, 1934, 28, 3068.Guglielmelli, Franco, *Chem. Abstracts*, 1931, 25, 4252.**4-Nitro-2-aminodiphenylamine** $\text{C}_{12}\text{H}_{11}\text{O}_2\text{N}_3$  MW, 229Red needles +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. 134° (125°). Sol. EtOH, AcOH. Mod. sol.  $\text{C}_6\text{H}_6$ . $\alpha\text{-N-Me deriv.}$ :  $\text{C}_{13}\text{H}_{11}\text{O}_2\text{N}_3$ . MW, 243. Red cryst. M.p. 76°. Sol. usual org. solvents.Ullmann, *Ann.*, 1904, 332, 98.Nietzki, Almenrader, *Ber.*, 1895, 28, 2971.Lindemann, *Ber.*, 1924, 57, 559.**6-Nitro-2-aminodiphenylamine.**

Dark red cryst. from EtOH. M.p. 101°.

Borsche, Rantscheff, *Ann.*, 1911, 379, 168.**2'-Nitro-2-aminodiphenylamine.**Yellowish-red needles from  $\text{H}_2\text{O}$ . M.p. 103°.Kehrmann, Steiner, *Ber.*, 1901, 34, 3091.**4'-Nitro-2-aminodiphenylamine.**Reddish-brown needles with blue lustre from EtOH.Aq. M.p. 144°. Sol. EtOH. Spar. sol.  $\text{C}_6\text{H}_6$ ,  $\text{Et}_2\text{O}$ .Ullmann, Dahmen, *Ber.*, 1908, 41, 3755.**2'-Nitro-3-aminodiphenylamine** $\text{C}_{12}\text{H}_{11}\text{O}_2\text{N}_3$  MW, 229Reddish needles from EtOH. M.p. 112°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , AcOH. Spar. sol.  $\text{H}_2\text{O}$ .Kehrmann, Steiner, *Ber.*, 1901, 34, 3090.**4'-Nitro-3-aminodiphenylamine.**Yellowish-brown plates from EtOH.Aq. M.p. 156°. Sol. EtOH,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{C}_6\text{H}_6$ . Insol. ligroin.Ullmann, Dahmen, *Ber.*, 1908, 41, 3754.**2-Nitro-4-aminodiphenylamine** $\text{C}_{12}\text{H}_{11}\text{O}_2\text{N}_3$  MW, 229

$\alpha$ -N-Et deriv.:  $C_{14}H_{15}O_2N_3$ . MW, 257.  
Cryst. M.p. 86°. B, HCl: orange leaflets from EtOH. M.p. 183-5° decomp.

Storrie, Tucker, *J. Chem. Soc.*, 1931, 2258.

### 2'-Nitro-4-aminodiphenylamine.

Cryst. M.p. 105-6°. Sol. hot EtOH.

Bandrowski, *Chem. Zentr.*, 1900, II, 852.

### 4'-Nitro-4-aminodiphenylamine.

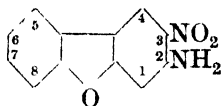
Reddish-brown needles from EtOH. M.p. 211-12° (205°). Sol. EtOH, Me<sub>2</sub>CO. Spar. sol. C<sub>6</sub>H<sub>6</sub>, Et<sub>2</sub>O. Insol. ligroin.

Morgan, Micklethwait, *J. Chem. Soc.*, 1908, 93, 611.

Ullmann, Dahmen, *Ber.*, 1908, 41, 3753.

Ullmann, D.R.P., 193,448, (*Chem. Zentr.*, 1908, I, 1003).

### 3-(?)-Nitro-2-aminodiphenylene oxide



$C_{12}H_8O_3N_2$  MW, 228

Orange-red needles with green reflex from PhNO<sub>2</sub> or toluene. M.p. 222°. Conc. H<sub>2</sub>SO<sub>4</sub> → light yellow sol. Mod. sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, AcOH. Spar. sol. EtOH, Et<sub>2</sub>O. There is some doubt as to the position of the nitro group.

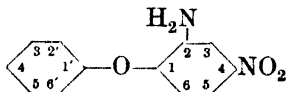
Borsche, Schacke, *Ber.*, 1923, 56, 2504.

### 6-Nitro-2-aminodiphenylene oxide.

Yellowish-orange needles from Me<sub>2</sub>CO-EtOH. M.p. 268°. Sol. hot AcOH, Me<sub>2</sub>CO. Slightly sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.

Cullinane, *J. Chem. Soc.*, 1932, 2367.

### 4-Nitro-2-aminodiphenyl Ether



$C_{12}H_{10}O_3N_2$  MW, 230

Red cryst. from EtOH. M.p. 107°.

N-Acetyl: m.p. 118°.

Brewster, Strain, *J. Am. Chem. Soc.*, 1934, 56, 118.

Ryan, Keane, M'Gahon, *Chem. Abstracts*, 1928, 22, 70.

### 5-Nitro-2-aminodiphenyl Ether.

Yellow plates from dil. MeOH. M.p. 116°.

N-Acetyl: yellow cryst. from MeOH. M.p. 180°.

McCombie, Macmillan, Scarborough, *J. Chem. Soc.*, 1931, 531.

Bayer, D.R.P., 228,763, (*Chem. Zentr.*, 1911, I, 105).

### 2'-Nitro-2-aminodiphenyl Ether.

Yellow needles from EtOH. M.p. 56°.

Cullinane, Davey, Padfield, *J. Chem. Soc.*, 1934, 719.

### 3-Nitro-4-aminodiphenyl Ether.

Red prisms from dil. MeOH. M.p. 82°.

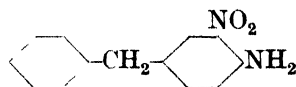
N-Acetyl: yellow needles from EtOH. M.p. 104° (100°).

Brewster, Strain, *J. Am. Chem. Soc.*, 1934, 56, 118.

Scarborough, *J. Chem. Soc.*, 1929, 2366.

Oesterlin, *Monatsh.*, 1931, 57, 38.

### 3-Nitro-4-aminodiphenylmethane



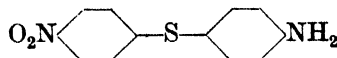
$C_{13}H_{12}O_2N_2$  MW, 228

Orange plates from MeOH, needles from dil. MeOH. M.p. 78°. Br in AcOH → 5-bromo-3-nitro-4-aminodiphenylmethane.

N-Acetyl: yellow plates from dil. MeOH. M.p. 99°.

Waters, *J. Chem. Soc.*, 1935, 1875.

### 4'-Nitro-4-aminodiphenyl sulphide



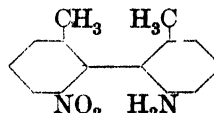
$C_{12}H_{10}O_2N_2S$  MW, 246

Orange prisms with blue reflex from C<sub>6</sub>H<sub>6</sub>. M.p. 143°. Sol. usual org. solvents. Insol. H<sub>2</sub>O.

Kehrmann, Bauer, *Ber.*, 1896, 29, 2362.

Fromm, Wittmann, *Ber.*, 1908, 41, 2264.

### 6'-Nitro-6-amino-2 : 2'-ditolyl (6'-Nitro-6-amino-2 : 2'-dimethyldiphenyl)



$C_{14}H_{14}O_2N_2$  MW, 242

d.

M.p. 122-3°.  $[\alpha]_D^{20} + 40^\circ$ .

d-Tartrate: m.p. 141°.

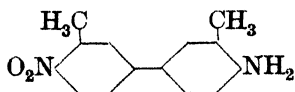
l.

Yellowish leaflets. M.p. 122-3°.  $[\alpha]_D^{20} - 40^\circ$ .

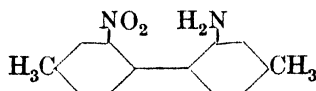
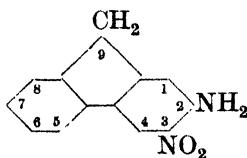
d-Tartrate: m.p. 151°.

dl.-

Yellow prisms or leaflets. M.p. 129-30°.

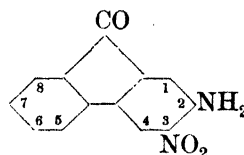
Angeletti, *Gazz. chim. ital.*, 1931, **61**, 651.**4'-Nitro-4-amino-3 : 3'-ditolyl** (4'-Nitro-4-amino-3 : 3'-dimethyldiphenyl) $C_{14}H_{14}O_2N_2$  MW, 242

Yellow needles from EtOH. M.p. 142-3°.

Schultz, Rohde, Vicari, *Ann.*, 1907, **352**, 121.**2'-Nitro-2-amino-4 : 4'-ditolyl** (2'-Nitro-2-amino-4 : 4'-dimethyldiphenyl) $C_{14}H_{14}O_2N_2$  MW, 242*B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 129-30°.*N-Acetyl*: light yellow cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 145-6°.*N-Diacetyl*: yellow cryst. from pet. ether. M.p. 116-17°.Mascarelli, Gatti, *Gazz. chim. ital.*, 1929, **59**, 858.**3-Nitro-2-aminofluorene** (3-Nitro-2-fluoryl-amine) $C_{13}H_{10}O_2N_2$  MW, 226Brownish-red needles from AcOH. M.p. 206°. Mod. sol. AcOH. Spar. sol. hot EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.*B.HCl*: bronze cryst. from AcOH + conc. HCl.Diels, Schill, Tolson, *Ber.*, 1902, **35**, 3286.**7-Nitro-2-aminofluorene** (7-Nitro-2-fluoryl-amine).

Orange-red prisms. M.p. 232°. Sol. EtOH, AcOH.

See previous reference.

**3-Nitro-2-aminofluorenone** $C_{13}H_8O_3N_2$  MW, 240Violet-red cryst. from PhNO<sub>2</sub>. M.p. 269°.*N-Acetyl*: red cryst. from chlorobenzene. M.p. 245-6°.*N-Carboethoxyl*: reddish-yellow cryst. from chlorobenzene. M.p. 204°.Eckert, Langecker, *J. prakt. Chem.*, 1928, **118**, 263.**7-Nitro-2-aminofluorenone.**

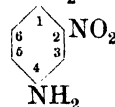
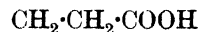
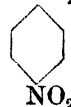
Needles from chlorobenzene. M.p. 279°.

*N-Acetyl*: red cryst. from PhNO<sub>2</sub>. Does not melt below 300°.

See previous reference.

**7-Nitro-4-aminofluorenone.**Scarlet micro-needles from Py. M.p. 292-3°. Sol. hot AcOEt, Py, aniline, dimethylaniline, chlorobenzene. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin, CCl<sub>4</sub>.Moore, Huntress, *J. Am. Chem. Soc.*, 1927, **49**, 1331.**Nitroaminoguaiacol.**

See under Nitroaminocatechol.

**2-Nitro-4-aminohydrocinnamic Acid** $C_9H_{10}O_4N_2$  MW, 210Red plates or needles from H<sub>2</sub>O. M.p. 137-9°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Insol. CS<sub>2</sub>.Gabriel, Zimmermann, *Ber.*, 1879, **12**, 601.**3-Nitro-4-aminohydrocinnamic Acid.**Orange cryst. M.p. 145°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. CS<sub>2</sub>.*N-Acetyl*: yellow needles. M.p. 174°. Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O, H<sub>2</sub>O. Insol. CS<sub>2</sub>.Gabriel, Steudemann, *Ber.*, 1882, **15**, 844.**p-Nitro- $\alpha$ -aminohydrocinnamic Acid** $C_9H_{10}O_4N_2$ 

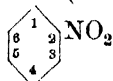
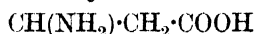
MW, 210

Prisms +  $1\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$  or  $\text{NH}_4\text{OH}$ . M.p.  $245^\circ$  decomp. after darkening at  $220^\circ$ .

*B,HCl*: needles from  $\text{H}_2\text{O}$ . M.p.  $220^\circ$  decomp. Sol.  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ .

Curtius, Mühlhäusser, *J. prakt. Chem.*, 1930, 125, 298.

## o-Nitro-β-aminohydrocinnamic Acid


 $\text{C}_9\text{H}_{10}\text{O}_4\text{N}_2$ 

MW, 210

Yellow plates from  $\text{H}_2\text{O}$ . M.p.  $222^\circ$  decomp. Sol. dil. acids and alkalis. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

Posner, *Ann.*, 1912, 389, 40.

## m-Nitro-β-aminohydrocinnamic Acid.

Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $236^\circ$  decomp. Sol. dil. acids and alkalis. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*B,HCl*: m.p.  $210\text{--}211.5^\circ$ .

Rodionow, Malewinskaja, *Ber.*, 1926, 59, 2952.

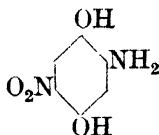
See also previous reference.

## p-Nitro-β-aminohydrocinnamic Acid.

Yellow cryst. powder. M.p.  $226\text{--}7^\circ$  decomp. Sol. dil. acids and alkalis. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

See previous references.

## 5-Nitro-2-aminohydroquinone


 $\text{C}_6\text{H}_6\text{O}_4\text{N}_2$ 

MW, 170

Red cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $154^\circ$  decomp. Dil. alkalis  $\rightarrow$  blue sol.  $\rightarrow$  green, then yellow on standing.

*Di-Me ether*:  $\text{C}_8\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 198. Yellow prisms from  $\text{C}_6\text{H}_6$ . M.p.  $158^\circ$ . *N-Acetyl*: yellow needles. M.p.  $164^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ .

*1-Me-4-Et ether*:  $\text{C}_9\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 212. Yellowish-brown cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $148^\circ$ .

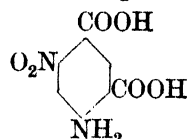
*Acetyl deriv.*: red needles from  $\text{EtOH}$ . M.p.  $226^\circ$  decomp.

*Diacetyl deriv.*: m.p.  $183\text{--}4^\circ$ .

Badische, D.R.P., 141,398, (*Chem. Zentr.*, 1903, I, 1163); D.R.P., 141,975, (*Chem. Zentr.*, 1903, I, 1380).

Heller, Hemmer, *J. prakt. Chem.*, 1931, 129, 207.

## 6-Nitro-4-aminoisophthalic Acid


 $\text{C}_8\text{H}_6\text{O}_6\text{N}_2$ 

MW, 226

Yellow prisms from  $\text{AcOH}\text{--}\text{EtOH}$ . M.p.  $280^\circ$  decomp. Spar. sol.  $\text{H}_2\text{O}$ .

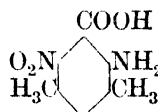
*Di-Me ester*:  $\text{C}_{10}\text{H}_{10}\text{O}_6\text{N}_2$ . MW, 254. Yellowish-brown cryst. M.p.  $153^\circ$ . Sol.  $\text{EtOH}$ .

*N-Acetyl*: cryst. from  $\text{EtOH}$  or  $\text{AcOH}$ . M.p.  $264^\circ$  decomp. Sol.  $\text{EtOH}$ . Spar. sol.  $\text{H}_2\text{O}$ .

Errera, Maltese, *Gazz. chim. ital.*, 1903, 33, ii, 287.

Bogert, Kropff, *J. Am. Chem. Soc.*, 1909, 31, 846.

## 6-Nitro-2-aminomesitylenic Acid (6-Nitro-2-amino-3:5-dimethylbenzoic acid)

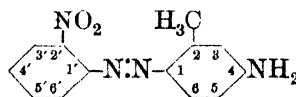

 $\text{C}_9\text{H}_{10}\text{O}_4\text{N}_2$ 

MW, 210

Yellow needles from  $\text{H}_2\text{O}$  or xylene, plates from  $\text{EtOH}$ . M.p.  $190^\circ$ . Easily sol.  $\text{EtOH}$ . Sol.  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ , hot xylene.

Bamberger, Demuth, *Ber.*, 1901, 34, 31.

## 2'-Nitro-4-amino-2-methylazobenzene


 $\text{C}_{13}\text{H}_{12}\text{O}_2\text{N}_4$ 

MW, 256

Violet needles from  $\text{EtOH}$ . M.p.  $119\text{--}21^\circ$ .

Mehner, *J. prakt. Chem.*, 1902, 65, 461.

## 3'-Nitro-4-amino-2-methylazobenzene.

Orange needles from  $\text{EtOH}$ . M.p.  $172^\circ$ .

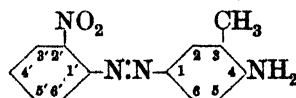
Mehner, *J. prakt. Chem.*, 1902, 65, 459.

## 4'-Nitro-4-amino-2-methylazobenzene.

Deep reddish-violet needles from  $\text{EtOH}$ . M.p.  $152\text{--}3^\circ$ .

Mehner, *J. prakt. Chem.*, 1902, 65, 457.

## 2'-Nitro-4-amino-3-methylazobenzene


 $\text{C}_{13}\text{H}_{12}\text{O}_2\text{N}_4$ 

MW, 256

Red needles from ligroin. M.p.  $99^\circ$ .

Mehner, *J. prakt. Chem.*, 1902, 65, 468.

**3'-Nitro-4-amino-3-methylazobenzene.**

Red plates from EtOH or  $C_6H_6$ . M.p. 151–2°.

Mehner, *J. prakt. Chem.*, 1902, **65**, 467.

**4'-Nitro-4-amino-3-methylazobenzene.**

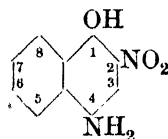
Reddish-brown cryst. from EtOH. M.p. 200–1° (195–7°).

Mehner, *J. prakt. Chem.*, 1902, **65**, 464.

A.G.F.A., D.R.P., 131,860, (*Chem. Zentr.*, 1902, II, 83).

**Nitroaminomethylnaphthalene.**

See Nitromethylnaphthylamine.

**2-Nitro-4-amino-1-naphthol**

$C_{10}H_8O_3N_2$  MW, 204

Maroon needles from EtOH. M.p. 160° decomp.

*B.HCl*: pale yellow needles from HCl-EtOH. M.p. 175° decomp.

*Me ether*:  $C_{11}H_{10}O_3N_2$ . MW, 218. *N-Acetyl*: needles. M.p. 214°.

*N-Acetyl*: yellow-orange needles from AcOH. M.p. 250° (238°) decomp.

*N-Benzoyl*: orange needles from EtOH. M.p. 330°.

Hodgson, Smith, *J. Chem. Soc.*, 1935, 673.

**5-Nitro-8-amino-1-naphthol.**

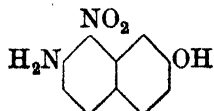
*Me ether*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Reddish-brown cryst. from EtOH. M.p. 193°. Spar. sol.  $H_2O$ .

*N-Acetyl*: red needles from EtOH.Aq. M.p. 240°. Sol. EtOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .

O : *N-Diacetyl*: needles from EtOH. M.p. 224°.

Fichter, Kühnel, *Ber.*, 1909, **42**, 4751.

Fichter, Gageur, *Ber.*, 1906, **39**, 3335.

**8-Nitro-7-amino-2-naphthol**

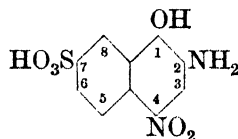
$C_{10}H_8O_3N_2$  MW, 204

*Me ether*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Reddish-yellow needles from  $H_2O$  or ligroin. M.p. 115–16°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Spar. sol. hot  $H_2O$ . *B.HBr*: yellow plates. M.p. 159–60°. *Picrate*: red needles from MeOH. M.p. 125°. *N-Acetyl*: yellow needles from EtOH. M.p. 149–50°. *N-Benzoyl*: yellowish

needles from AcOH. M.p. 203–4°. *N-Benzylidene*: reddish cryst. from  $Et_2O$ -pet. ether. M.p. 126–7°. *N-Salicylidene*: light brown needles from ligroin. M.p. 202°.

O : *N-Di-Me*:  $C_{12}H_{12}O_3N_2$ . MW, 232. Yellowish-red needles from ligroin. M.p. 149–50°. Sol. MeOH, EtOH,  $Et_2O$ . Spar. sol. ligroin. Almost insol.  $H_2O$ .  $HCl \rightarrow$  dark yellow sol.

Fischer, Kern, *J. prakt. Chem.*, 1916, **94**, 38.

**4-Nitro-2-amino-1-naphthol-7-sulphonic Acid**

$C_{10}H_8O_6N_2S$  MW, 284

Yellow cryst. Spar. sol. cold  $H_2O$ .

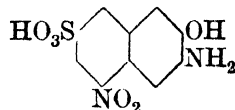
Ges. für. chem. Ind., D.R.P., 189,513, (*Chem. Zentr.*, 1907, II, 2006).

**2-Nitro-4-amino-1-naphthol-7-sulphonic Acid.**

Golden-yellow leaflets from  $H_2O$ . Mod. sol. hot  $H_2O$ . Alkalis  $\rightarrow$  intense blood-red sols.

Lauterbach, *Ber.*, 1881, **14**, 2029.

See also previous reference.

**5-Nitro-3-amino-2-naphthol-7-sulphonic Acid**

$C_{10}H_8O_6N_2S$  MW, 284

Yellow needles. Mod. sol.  $H_2O$ .

Cassella, D.R.P., 110,369, (*Chem. Zentr.*, 1900, II, 548).

**5-Nitro-2-aminonicotinic Acid (5-Nitro-2-aminopyridine-3-carboxylic acid)**

$C_6H_5O_4N_3$  MW, 183

Golden-yellow needles from  $H_2O$  or  $C_6H_6$ . M.p. 233°. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ .

Räth, *Ann.*, 1931, **486**, 294.

**5-Nitro-6-aminonicotinic Acid (5-Nitro-6-aminopyridine-3-carboxylic acid).**

Yellow needles from  $H_2O$ . M.p. 300–1° (280°) decomp. Sol. hot  $H_2O$ , EtOH. Spar. sol. usual

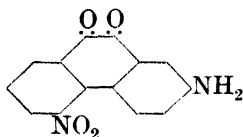


org. solvents. Mod. sol. acids. Sublimes with part. decomp.

Marckwald, *Ber.*, 1893, **26**, 2189; 1894, **27**, 1334.

Räth, Prange, *Ann.*, 1928, **467**, 8.

### 5-Nitro-2-aminophenanthraquinone



$C_{14}H_8O_4N_2$

MW, 268

Dark brown powder. Decomp. on heating. Spar. sol.  $H_2O$  and common org. solvents. With  $H_2SO_4 \rightarrow$  dark brown sol. With  $NaOH.Aq. \rightarrow$  yellowish-green sol. Constitution given as 4-nitro-5-aminophenanthraquinone until 1926.

*N*-Diacetyl: brown needles. M.p.  $280^\circ$ .

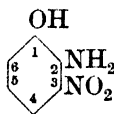
Schmidt, Leipprand, *Ber.*, 1905, **38**, 3735.

Christie, Holderness, Kenner, *J. Chem. Soc.*, 1926, 671.

### Nitroaminophenetole.

See Nitrophenetidine.

### 3-Nitro-*o*-aminophenol



$C_6H_6O_3N_2$

MW, 154

Red needles from  $H_2O$ . M.p.  $216-17^\circ$  ( $212^\circ$ ). Sol. 100 parts boiling  $H_2O$ .

*Me ether*: see 3-Nitro-*o*-anisidine.

*Et ether*: see 3-Nitro-*o*-phenetidine.

*N*-Acetyl: 6-nitro-2-hydroxyacetanilide. Yellow cryst. M.p.  $172^\circ$ . *O*-*p*-Toluenesulphonyl: m.p.  $120^\circ$ .

*O*-*p*-Toluenesulphonyl: pale yellow cryst. M.p.  $136^\circ$ .

King, *J. Chem. Soc.*, 1927, 1058.

Fourneau, Tréfouel, Tréfouel, *Bull. soc. chim.*, 1927, **41**, 448.

### 4-Nitro-*o*-aminophenol.

Orange prisms +  $H_2O$ . M.p.  $80-90^\circ$ , anhyd.  $142-3^\circ$ . Sol.  $EtOH$ ,  $Et_2O$ . Spar. sol.  $H_2O$ .  $k = 0.26 \times 10^{-6}$  at  $25^\circ$ .

*Me ether*: see 4-Nitro-*o*-anisidine.

*Et ether*: see 4-Nitro-*o*-phenetidine.

*N*-Benzoyl: needles from aniline. Decomp. above  $200^\circ$ . Spar. sol.  $EtOH$ ,  $AcOH$ .

*O*-*p*-Toluenesulphonyl: yellowish needles from  $AcOH$  or  $EtOH$ . M.p.  $122^\circ$ .

Post, Stuckenberg, *Ann.*, 1880, **205**, 72.

Hofer, Jakob, *Ber.*, 1908, **41**, 3196.

### 5-Nitro-*o*-aminophenol.

Light brown needles from  $H_2O$ . M.p.  $201-2^\circ$ .

*Me ether*: see 5-Nitro-*o*-anisidine.

*Et ether*: see 5-Nitro-*o*-phenetidine.

*O*: *N*-Diacetyl: needles from  $H_2O$ . M.p.  $187^\circ$ .

*N*-Anisylidene: yellow plates from  $C_6H_6$ . M.p.  $160-1^\circ$ . Sol.  $C_6H_6$ . Insol. ligroin.

*O*-*p*-Toluenesulphonyl: light yellow cryst. from  $EtOH$  or  $AcOH$ . M.p.  $188^\circ$  ( $185^\circ$ ). *N*-Acetyl: m.p.  $189^\circ$ .

Meldola, Woolcott, Wray, *J. Chem. Soc.*, 1896, **69**, 1325.

A.G.F.A., D.R.P., 165,650, (*Chem. Zentr.*, 1906, **1**, 516).

### 6-Nitro-*o*-aminophenol.

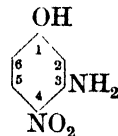
Red needles from  $EtOH.Aq$ . M.p.  $111-12^\circ$ . Very sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $AcOH$ . Sol.  $EtOH$ . Spar. sol.  $H_2O$ .

*Me ether*: see 6-Nitro-*o*-anisidine.

Benda, *Ber.*, 1914, **47**, 1010.

Post, Stuckenberg, *Ann.*, 1880, **205**, 85.

### 4-Nitro-*m*-aminophenol



$C_6H_6O_3N_2$

MW, 154

Orange needles from  $H_2O$ . M.p.  $185-6^\circ$ .

*Me ether*: see 4-Nitro-*m*-anisidine.

*Et ether*: see 4-Nitro-*m*-phenetidine.

*N*-Acetyl: 6-nitro-3-hydroxyacetanilide. Needles from  $AcOH$ . M.p.  $266^\circ$ .

*N*-Phenyl: see 6-Nitro-3-hydroxydiphenylamine.

Meldola, Stephens, *J. Chem. Soc.*, 1906, **89**, 924.

### 5-Nitro-*m*-aminophenol.

Yellow cryst. M.p.  $165^\circ$ . Very sol.  $EtOH$ ,  $Et_2O$ ,  $AcOH$ ,  $Me_2CO$ . Spar. sol.  $C_6H_6$ ,  $CHCl_3$ , ligroin.

*Me-ether*: see 5-Nitro-*m*-anisidine.

*Et ether*: see 5-Nitro-*m*-phenetidine.

*N*-Acetyl: 5-nitro-3-hydroxyacetanilide. Dark yellow prisms from  $AcOEt$  or 50%  $AcOH$ . M.p.  $260-70^\circ$  decomp. Sol.  $EtOH$ ,  $Me_2CO$ ,  $AcOH$ . Spar. sol.  $C_6H_6$ ,  $Et_2O$ ,  $CHCl_3$ , ligroin.

Heller, *Ber.*, 1909, **42**, 2193.

**6-Nitro-*m*-aminophenol.**

Orange-yellow needles from  $\text{H}_2\text{O}$ . M.p.  $162^\circ$  ( $158^\circ$ ).

*Me ether*: see 6-Nitro-*m*-anisidine.

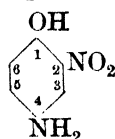
*Et ether*: see 6-Nitro-*m*-phenetidine.

*N-Acetyl*: 4-nitro-3-hydroxyacetanilide. Yellow prisms or needles from  $\text{AcOH}$ . M.p.  $221^\circ$ .

*O:N-Diacetyl*: needles from  $\text{H}_2\text{O}$  or  $\text{AcOH}$ . M.p.  $149^\circ$ . Sol.  $\text{EtOH}$ .

M.L.B., D.R.P., 285,638, (*Chem. Zentr.*, 1915, II, 511).

Meldola, Stephens, *J. Chem. Soc.*, 1906, 89, 925.

**2-Nitro-*p*-aminophenol**

$\text{C}_6\text{H}_6\text{O}_3\text{N}_2$

MW, 154

Dark red plates or needles. M.p.  $131^\circ$  ( $127^\circ$ ).

*Me ether*: see 2-Nitro-*p*-anisidine.

*Et ether*: see 2-Nitro-*p*-phenetidine.

*N-Me*:  $\text{C}_7\text{H}_8\text{O}_3\text{N}_2$ . MW, 168. Reddish-brown cryst. from  $\text{EtOH}$ . M.p.  $113-14^\circ$ .

*N-Acetyl*: 3-nitro-4-hydroxyacetanilide. Yellow needles. M.p.  $157-8^\circ$ .

Bart, D.R.P., 258,059, (*Chem. Zentr.*, 1913, I, 1374).

Friedländer, Zeitlin, *Ber.*, 1894, 27, 196.

Girard, *Bull. soc. chim.*, 1924, 35, 772.

**3-Nitro-*p*-aminophenol.**

Dark red prisms with green reflex from  $\text{Et}_2\text{O}$ . M.p.  $154^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

*Me ether*: see 3-Nitro-*p*-anisidine.

*Et ether*: see 3-Nitro-*p*-phenetidine.

*N-Acetyl*: 2-nitro-4-hydroxyacetanilide. Brownish-yellow needles from  $\text{H}_2\text{O}$ . M.p.  $218^\circ$ .

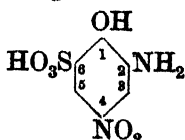
*O-m-Nitrobenzoyl*: yellow needles. M.p.  $184^\circ$ .

*O-p-Toluenesulphonyl*: yellow plates from  $\text{EtOH}$ . M.p.  $134^\circ$ .

*O:N-Diacetyl*: yellow prisms from  $\text{EtOH}$ . Aq. M.p.  $146-7^\circ$ .

Reverdin, *Ber.*, 1906, 39, 3796.

Hähle, *J. prakt. Chem.*, 1891, 43, 64.

**4-Nitro-*o*-aminophenol-6-sulphonic Acid**

$\text{C}_6\text{H}_6\text{O}_6\text{N}_2\text{S}$

MW, 234

Dict. of Org. Comp.—III.

Prisms +  $1\text{H}_2\text{O}$ , needles +  $2\text{H}_2\text{O}$ . M.p.  $285^\circ$  decomp.

King, *J. Chem. Soc.*, 1921, 119, 1415.

Badische, D.R.P., 123,611, (*Chem. Zentr.*, 1901, II, 797).

**5-Nitro-*o*-aminophenol-4-sulphonic Acid.**

Yellow cryst. Sol.  $\text{H}_2\text{O}$ .

M.L.B., D.R.P., 188,378, (*Chem. Zentr.*, 1907, II, 1467).

**6-Nitro-*o*-aminophenol-4-sulphonic Acid.**

Prisms. Sol. hot  $\text{H}_2\text{O}$  with reddish-yellow col. Spar. sol.  $\text{EtOH}$  with yellow col.  $\text{FeCl}_3 \rightarrow$  greenish-yellow col.

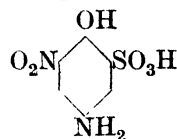
*Na salt*: reddish-brown needles. Very sol.  $\text{H}_2\text{O}$  with red col.

*K salt*: brown needles from  $\text{H}_2\text{O}$ . Sol. hot  $\text{H}_2\text{O}$  with brownish-yellow col.

Badische, D.R.P., 121,427, (*Chem. Zentr.*, 1901, I, 1396).

M.L.B., D.R.P., 148,213, (*Chem. Zentr.*, 1904, I, 414).

Hillyer, U.S.P., 1,504,044, (*Chem. Abstracts*, 1924, 18, 3194).

**6-Nitro-*p*-aminophenol-2-sulphonic Acid**

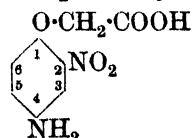
$\text{C}_6\text{H}_6\text{O}_6\text{N}_2\text{S}$

MW, 234

Reddish-brown needles from  $\text{H}_2\text{O}$ . Does not melt below  $290^\circ$ . Spar. sol. hot  $\text{H}_2\text{O}$ . Insol.  $\text{EtOH}$ .

Badische, D.R.P., 113,337, (*Chem. Zentr.*, 1900, II, 656).

King, *J. Chem. Soc.*, 1921, 119, 1415.

**2-Nitro-4-aminophenoxyacetic Acid**

$\text{C}_8\text{H}_8\text{O}_5\text{N}_2$

MW, 212

Brownish-yellow prisms from  $\text{H}_2\text{O}$ . M.p.  $196^\circ$ .

*N-Acetyl*: reddish-yellow needles from  $\text{H}_2\text{O}$ . M.p.  $205^\circ$ .

Howard, *Ber.*, 1897, 30, 2106.

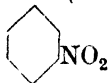
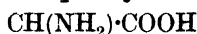
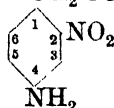
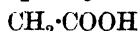
Girard, *Bull. soc. chim.*, 1924, 35, 772.

**3-Nitro-4-aminophenoxyacetic Acid.**

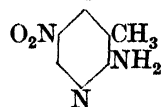
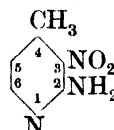
Brown needles. M.p. 185°.

N-Acetyl: cryst. from H<sub>2</sub>O. M.p. 174°.

N-Benzoyl: yellow needles from AcOH. M.p. 176-7°. Sol. warm EtOH, AcOH.

See second reference above and also Reverdin, *Ber.*, 1909, 42, 4113.**3-Nitro-α-aminophenylacetic Acid**C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub> MW, 196Needles. M.p. 172° decomp. Sol. H<sub>2</sub>O. Insol. EtOH. Easily sol. HCl.Plöchl, Loë, *Ber.*, 1885, 18, 1179.**2-Nitro-4-aminophenylacetic Acid**C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub> MW, 196Reddish-brown needles. M.p. 184-6°. Sol. H<sub>2</sub>O, EtOH. Mod. sol. Et<sub>2</sub>O. Insol. CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. sol. acids, alk.Me ester: C<sub>9</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 210. M.p. 96°.Et ester: C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub>. MW, 224. Yellow needles. M.p. 100°.Gabriel, Meyer, *Ber.*, 1881, 14, 824.**3-Nitro-4-aminophenylacetic Acid.**Orange-yellow plates or needles from H<sub>2</sub>O. M.p. 143.5-144.5°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, AcOH. Mod. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. CS<sub>2</sub>.Gabriel, *Ber.*, 1882, 15, 836.**3-Nitro-6-amino-α-picoline**C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub> MW, 153M.p. 188°. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Very spar. sol. H<sub>2</sub>O. Dil. acids → yellow sols. Non-volatile in steam.Seide, *Chem. Zentr.*, 1923, 3, 1022.**5-Nitro-6-amino-α-picoline.**M.p. 141°. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Insol. EtOH. Dil. acids → yellow sols. Volatile in steam.

See previous reference.

**5-Nitro-2-amino-β-picoline**C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub> MW, 153Yellow prisms from EtOH. M.p. 255°. Sol. hot EtOH. Spar. sol. H<sub>2</sub>O. Sol. min. acids with yellow col.Seide, *Ber.*, 1924, 57, 1805.**3-Nitro-2-amino-γ-picoline**C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub> MW, 153Yellow needles from H<sub>2</sub>O. M.p. 136°. Sol. H<sub>2</sub>O, EtOH, acids. Volatile in steam.Seide, *Ber.*, 1924, 57, 794.**5-Nitro-2-amino-γ-picoline.**Yellow prisms from EtOH. M.p. 220°. Mod. sol. EtOH. Spar. sol. H<sub>2</sub>O.

See previous reference.

**3-Nitro-2-aminopyridine**C<sub>5</sub>H<sub>5</sub>O<sub>2</sub>N<sub>3</sub> MW, 139

Yellow needles. M.p. 164°.

N-Me: C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub>. MW, 153. Golden-yellow plates. M.p. 63-4°. B.p. 262-262.5°/740 mm. decomp. Sol. H<sub>2</sub>O, usual org. solvents.N-NO<sub>2</sub>: 3-nitropyridyl-2-nitramine. C<sub>5</sub>H<sub>4</sub>O<sub>4</sub>N<sub>4</sub>. MW, 184. Yellow plates from H<sub>2</sub>O. M.p. 137°.Tschitschibabin, Kirssanow, *Ber.*, 1928, 61, 1228.Chichibabin, D.R.P., 374,291, (*Chem. Abstracts*, 1924, 18, 2176). Note: this is the alternative spelling of the author's name given in Chem. Abstracts.**5-Nitro-2-aminopyridine.**

Yellow leaflets. M.p. 188°.

N-Me: cryst. M.p. 181°.

N-NO<sub>2</sub>: 5-nitropyridyl-2-nitramine. Cryst. from H<sub>2</sub>O. Decomp. on heating. Sol. dil. alkalis. Insol. dil. acids.

See previous references.

**3-Nitro-4-aminopyridine.**

Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $200^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH.

$B, \text{HCl}$ : prisms. M.p.  $258-9^\circ$ . Easily decomp.

$\text{B}_2, \text{H}_2\text{PtCl}_6$ : yellow plates. M.p.  $256^\circ$ .

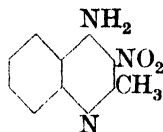
*Picrate*: needles. M.p.  $197-8^\circ$ .

Koenigs, Miels, Gurlt, *Ber.*, 1924, **57**, 1183.

Koenigs, Freter, *ibid.*, 1190.

**N-Nitroaminopyridine.**

See Pyridylnitramine.

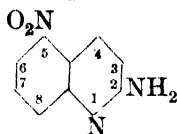
**3-Nitro-4-aminoquinaldine**

$\text{C}_{10}\text{H}_9\text{O}_2\text{N}_3$

MW, 203

Pale yellow needles. M.p.  $201^\circ$ . Sublimes.

Conrad, Limpach, *Ber.*, 1888, **21**, 1982.

**5-Nitro-2-aminoquinoline**

$\text{C}_9\text{H}_7\text{O}_2\text{N}_3$

MW, 189

Orange prisms from toluene. M.p.  $239^\circ$ . Sol. EtOH. Mod. sol.  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ .

Fischer, Guthmann, *J. prakt. Chem.*, 1916, **93**, 378.

**6-Nitro-2-aminoquinoline.**

Yellow prisms from toluene. M.p.  $265^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ , Py. Mod. sol.  $\text{C}_6\text{H}_6$ .

See previous reference.

**8-Nitro-2-aminoquinoline.**

Yellow cryst. from toluene. M.p.  $159^\circ$ . Sol. Py. Mod. sol. EtOH,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ .

$\text{B, HgCl}_2$ : yellow cryst. powder. M.p.  $216^\circ$ .

*Picrate*: orange cryst. M.p.  $257^\circ$ .

*N-Acetyl*: needles from AcOH.Aq. M.p.  $211^\circ$ . Very sol. AcOH. Mod. sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*N-Benzoyl*: needles from EtOH.Aq. M.p.  $166^\circ$ . Sol.  $\text{C}_6\text{H}_6$ , AcOH, Py. Mod. sol.  $\text{Et}_2\text{O}$ .

See previous reference.

**6-Nitro-4-aminoquinoline.**

Yellow needles from EtOH. M.p.  $272^\circ$  decomp. Sol. dil. min. acids. Insol. alkalis.  $\text{KMnO}_4 \rightarrow$  5-nitro-anthranilic acid.

*N-NO\_2*: 6-nitro-4-quinolylnitramine

$\text{C}_9\text{H}_6\text{O}_4\text{N}_4$ . MW, 234. Golden-yellow needles from 50% EtOH. Decomp. at  $203^\circ$ . Sol. alkalis.

Tschitschibabin, Witkovsky, Lapschin, *Ber.*, 1925, **58**, 807.

**8-Nitro-5-aminoquinoline.**

Needles. M.p.  $280^\circ$ . Sol. warm  $\text{H}_2\text{O}$ , EtOH. Almost insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether.

$B, \text{HCl}$ : yellow needles. M.p. about  $250^\circ$  decomp.

Dikshoorn, *Rec. trav. chim.*, 1929, **48**, 237.

**5-Nitro-6-aminoquinoline.**

Yellow needles from toluene. M.p.  $178^\circ$  ( $173-4^\circ$ ).

*N-p-Toluenesulphonyl*: yellow cryst. from EtOH. M.p.  $168^\circ$ .

*Picrate*: m.p.  $270^\circ$ .

Bryd, *Chem. Zentr.*, 1932, I, 3066.

Kaufmann, Zeller, *Ber.*, 1917, **50**, 1629.

**8-Nitro-7-aminoquinoline.**

Cryst. M.p.  $194^\circ$ .

Fourneau, Tréfouel, Tréfouel, Benoit, *Chem. Abstracts*, 1932, **26**, 1592.

**5-Nitro-8-aminoquinoline.**

Orange-red plates or needles from  $\text{C}_6\text{H}_6$ . M.p.  $197^\circ$ . Sol. EtOH. Less sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol. hot  $\text{H}_2\text{O}$ . Insol. pet. ether.

Dikshoorn, *Rec. trav. chim.*, 1929, **48**, 517.

Slater, *J. Chem. Soc.*, 1931, 1940.

**6-Nitro-8-aminoquinoline.**

Red cryst. from EtOH. M.p.  $194^\circ$ .

$\text{B}_2, \text{H}_2\text{PtCl}_6$ : red cryst. Decomp. at  $180^\circ$ .

*Methiodide*: red needles. M.p.  $176^\circ$ . Sol. hot EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ .

*N-Acetyl*: light yellow needles. M.p.  $224^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ , EtOH. Sublimes.

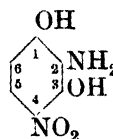
Claus, Hartmann, *J. prakt. Chem.*, 1896, **53**, 207.

Kaufmann, Hüsey, *Ber.*, 1908, **41**, 1740.

**N-Nitroaminoquinoline.**

See Quinolylnitramine.

**4-Nitro-2-aminoresorcinol** (3-Nitro-2:6-dihydroxyaniline)



$\text{C}_6\text{H}_6\text{O}_4\text{N}_2$

MW, 170

Brown cryst. from EtOH.Aq. M.p.  $182^\circ$ .

## 6-Nitro-4-aminoresorcinol

Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Reduces cold NH<sub>3</sub>.AgNO<sub>3</sub>.

Benedikt, v. Hubl, *Monatsh.*, 1881, 2, 324.

Heller, Lindner, Georgi, *Ber.*, 1923, 56, 1868.

**6-Nitro-4-aminoresorcinol** (5-Nitro-2:4-dihydroxyaniline).

Red cryst. from Et<sub>2</sub>O-ligroin. M.p. 160–1° decomp. Very sol. EtOH, Me<sub>2</sub>CO, Et<sub>2</sub>O. Less sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, ligroin. Sol. acids, alkalis.

*Di-Me ether*: C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 198. Red needles from EtOH. M.p. 136–7°. Sol. Me<sub>2</sub>CO. Mod. sol. EtOH. *N-Acetyl*: pale yellow needles from EtOH. M.p. 173°. Sol. hot AcOH. Mod. sol. Me<sub>2</sub>CO.

*N-Acetyl*: yellow needles from EtOH or AcOH. M.p. 261° decomp. Very sol. Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, ligroin.

*Triacetyl deriv.*: plates from toluene. M.p. 176° decomp. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOH, AcOEt. Spar. sol. C<sub>6</sub>H<sub>6</sub>, Et<sub>2</sub>O. Insol. ligroin.

Vermeulen, *Rec. trav. chim.*, 1919, 38, 110.

Heller, Sourlis, *Ber.*, 1910, 43, 2583.

## 5-Nitro-3-aminosalicylic Acid



C<sub>7</sub>H<sub>6</sub>O<sub>5</sub>N<sub>2</sub> MW, 198

Red needles + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 234° decomp. Sol. EtOH. Less sol. H<sub>2</sub>O. NH<sub>3</sub> → deep red sol. HCl → yellow sol.

Hübner, Babcock, Schaumann, *Ber.*, 1879, 12, 1345.

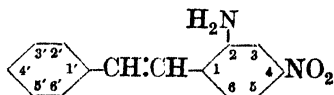
Meldola, Foster, Brightman, *J. Chem. Soc.*, 1917, 111, 540.

## 3-Nitro-5-aminosalicylic Acid.

Plates from H<sub>2</sub>O. M.p. 240° decomp. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

Cassella, D.R.P., 85,989.

## 4-Nitro-2-aminostilbene



C<sub>14</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub> MW, 240

Red cryst. from EtOH. M.p. 142–3°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>.

*B,HCl*: yellow cryst. from AcOH or HCl. M.p. 218–19°. Decomp. by H<sub>2</sub>O.

*N-Acetyl*: light yellow needles from EtOH. M.p. 220°.

Thiele, Escales, *Ber.*, 1901, 34, 2845.

## 84 2-Nitro-4'-aminostilbene-4-carboxylic Acid

### 2'-Nitro-2-aminostilbene.

Amorph. Easily decomp.

Bischoff, *Ber.*, 1888, 21, 2077.

### 2-Nitro-4-aminostilbene.

Dark red cryst. from EtOH. M.p. 110–11°.

*B,HCl*: golden-yellow cryst. M.p. 223°.

*N-Acetyl*: orange plates. M.p. 192–3°.

Thiele, Escales, *Ber.*, 1901, 34, 2846.

### 4'-Nitro-4-aminostilbene.

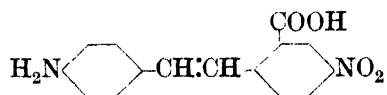
Purplish-red plates from PhNO<sub>2</sub>. M.p. 229–30°. Sol. hot PhNO<sub>2</sub>. Spar. sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*B,HCl*: yellow needles.

*N-Di-Me*: C<sub>16</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>. MW, 268. Red plates from C<sub>6</sub>H<sub>6</sub>. M.p. 250–1°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH.

Strakosch, *Ber.*, 1873, 6, 329.

## 4-Nitro-4'-aminostilbene-2-carboxylic Acid



C<sub>15</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub> MW, 284

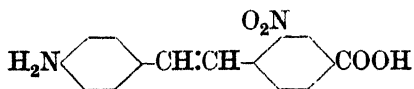
*N-Di-Me*: C<sub>17</sub>H<sub>16</sub>O<sub>4</sub>N<sub>2</sub>. MW, 312. Red cryst. from AcOH. M.p. 206° decomp. Sol. EtOH, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Very spar. sol. Et<sub>2</sub>O, ligroin.

*Me ester*: C<sub>18</sub>H<sub>18</sub>O<sub>4</sub>N<sub>2</sub>. MW, 326. Red cryst. from MeOH. M.p. 158–9°.

Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH. Sol. C<sub>6</sub>H<sub>6</sub> with golden-orange fluor. *Et ester*: C<sub>19</sub>H<sub>20</sub>O<sub>4</sub>N<sub>2</sub>. MW, 340. Red needles from EtOH. M.p. 139–40°. Sol. C<sub>6</sub>H<sub>6</sub> with strong golden-orange fluor.

Pfeiffer, Engelhardt, Alfuss, *Ann.*, 1928, 467, 176.

## 2-Nitro-4'-aminostilbene-4-carboxylic Acid



C<sub>15</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub> MW, 284

Brownish-yellow leaflets from AcOH. M.p. 255–63°.

*Me ester*: C<sub>16</sub>H<sub>14</sub>O<sub>4</sub>N<sub>2</sub>. MW, 298. Red needles from EtOH. M.p. 161°.

*Et ester*: C<sub>17</sub>H<sub>16</sub>O<sub>4</sub>N<sub>2</sub>. MW, 312. Red leaflets from EtOH. M.p. 134°.

*Nitrile*: C<sub>15</sub>H<sub>11</sub>O<sub>2</sub>N<sub>3</sub>. MW, 265. Red leaflets from EtOH. M.p. 202°. *Acetyl*: golden-yellow needles + C<sub>2</sub>H<sub>5</sub>OH from EtOH. M.p. 245°. *Benzoyl*: yellow cryst. from AcOH. M.p. 242°.

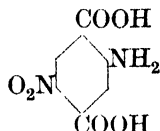
*N-Di-Me*: *Me ester*, orange leaflets from MeOH. M.p. 134°. *Et ester*: violet needles or yellow leaflets from EtOH. M.p. 129–30°.

*N-Acetyl*: golden-yellow powder from AcOH. M.p. 275°.

*N-Benzoyl*: orange cryst. from AcOH. M.p. 297°.

Pfeiffer, Kalckbrenner, Behr, *J. prakt. Chem.*, 1925, 109, 220.

## 5-Nitro-2-aminoterephthalic Acid

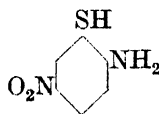


$C_8H_6O_6N_2$  MW, 226

Pale yellow cryst. Decomp. about 260°. Sol. hot  $H_2O$ , hot EtOH.

*Di-Me ester*:  $C_{10}H_{10}O_6N_2$ . MW, 254. Pale yellow prisms from EtOH. M.p. 187°. Sol. Py. Spar. sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . *N-Acetyl*: pale yellow prisms from EtOH. M.p. 142°. Very sol.  $CHCl_3$ . Sol. EtOH,  $Me_2CO$ , AcOH,  $C_6H_6$ , Py. Spar. sol.  $Et_2O$ ,  $CS_2$ , ligroin.

Kauffmann, Weissel, *Ann.*, 1912, 393, 16.

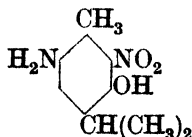
5-Nitro-*o*-aminothiophenol

$C_6H_6O_2N_2S$  MW, 170

Orange-yellow plates from EtOH.Aq., pale yellow needles from AcOH. M.p. 84°. Alkalis  $\rightarrow$  brownish-red sols.

Jacobsen, Kwaysser, *Ann.*, 1893, 277, 242.

## 2-Nitro-6-aminothymol



$C_{10}H_{14}O_3N_2$  MW, 210

Reddish-brown needles from EtOH. Very sol. EtOH.

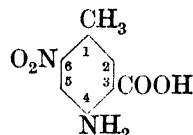
*Et ether*:  $C_{12}H_{18}O_3N_2$ . MW, 238. Plates from EtOH. M.p. 111–12°. Sol. EtOH,  $CS_2$ . *B.HCl*: needles. Decomp. at 200°. *N-Acetyl*: needles from EtOH.Aq. M.p. 119°. *N-Benzoyl*: needles from EtOH.Aq. M.p. 138°. Spar. sol.  $Et_2O$ .

*N-Benzoyl*: yellow needles from ligroin. M.p. 158–60°. Sol. EtOH,  $C_6H_6$ , toluene. Spar. sol. ligroin.

*O*: *N-Diacetyl*: yellow prisms from  $C_6H_6$ . M.p. 157–9°.

Soderi, *Gazz. chim. ital.*, 1895, 25, ii, 404.

Gaebel, *Ber.*, 1902, 35, 2794.

6-Nitro-4-amino-*m*-toluic Acid

$C_8H_8O_4N_2$  MW, 196

Red cryst. from EtOH or AcOEt. M.p. 239–40° decomp. Sol.  $H_2O$ . Spar. sol.  $C_6H_6$ . Alk. salts are yellow.

*Me ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Red cryst. M.p. 128°. Sol. EtOH.

*N*-2:4-Dinitrophenyl:  $C_{14}H_{10}O_8N_4$ . MW, 362. Yellow needles from AcOH. M.p. 298°. Spar. sol. common org. solvents.

*N-Acetyl*:  $C_{10}H_{10}O_5N_2$ . MW, 238. Yellow triclinic cryst. M.p. 223–5° decomp. Spar. sol.  $H_2O$ ,  $C_6H_6$ , xylene. Sol. EtOH, AcOH, AcOEt.

Errera, Maltese, *Gazz. chim. ital.*, 1905, 35, 370, 378.

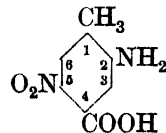
4-Nitro-6-amino-*m*-toluic Acid.

Yellow needles from  $H_2O$ . M.p. about 235°. Sol. EtOH, AcOH. Spar. sol.  $C_6H_6$ . Alk. salts are dark yellow.  $H_2SO_4$  at 200°  $\rightarrow$  4-nitro-*o*-toluidine.

*Me ester*: pale yellow needles from EtOH. M.p. 169°.

*N-Acetyl*: pale yellow plates from EtOH. M.p. 254–5° decomp.

See previous reference.

5-Nitro-2-amino-*p*-toluic Acid

$C_8H_8O_4N_2$  MW, 196

Yellowish-red needles from  $H_2O$ . M.p. 220° decomp. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

Claus, Beysen, *Ann.*, 1891, 266, 235.

6-Nitro-2-amino-*p*-toluic Acid.

Lemon-yellow needles from  $H_2O$ . M.p. 214°. Sol. EtOH,  $Et_2O$ . Sublimes.

*Na salt*: yellow needles +  $\frac{1}{2}H_2O$ .

*Mg salt*: yellow cryst. + 5H<sub>2</sub>O.

*Ba salt*: golden-yellow plates + 4H<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

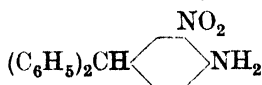
See previous reference.

### 6-Nitro-3-amino-*p*-toluic Acid.

Needles from EtOH. M.p. 235–6°. Spar. sol. H<sub>2</sub>O. Heat with HCl in sealed tube to 150° → 6-nitro-*m*-toluidine.

Fileti, Crosa, *Gazz. chim. ital.*, 1888, 18, 303.

### 3-Nitro-4-aminotriphenylmethane



C<sub>19</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub> MW, 304

Golden-yellow cryst. from EtOH.Aq. M.p. 98°. Very sol. most org. solvents.

Thomae, *J. prakt. Chem.*, 1905, 71, 568.

### Nitroaminoveratrol.

See under Nitroaminocatechol.

### 2-Nitro-*n*-amyl Alcohol (2-Nitropentanol-1)



C<sub>5</sub>H<sub>11</sub>O<sub>3</sub>N MW, 133

Liq. B.p. 130–6°/28 mm.

Jones, Kenner, *J. Chem. Soc.*, 1930, 926.

### 1-Nitro-*sec*-.*n*-amyl Alcohol (1-Nitro-2-pentanol)



C<sub>5</sub>H<sub>11</sub>O<sub>3</sub>N MW, 133

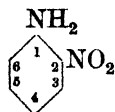
B.p. 117°/17 mm., 87–8°/3 mm. D<sub>4</sub><sup>20</sup> 1.0847. n<sub>D</sub><sup>20</sup> 1.4421.

*Acetyl*: b.p. 111–13°/10 mm. D<sub>4</sub><sup>20</sup> 1.0898. n<sub>D</sub><sup>20</sup> 1.4339.

See previous reference and also

Schmidt, Rutz, *Ber.*, 1928, 61, 2145.

### *o*-Nitroaniline



C<sub>6</sub>H<sub>6</sub>O<sub>2</sub>N<sub>2</sub> MW, 138

Golden-yellow plates or needles from H<sub>2</sub>O. M.p. 71.5°. Very sol. Et<sub>2</sub>O. Sol. EtOH, CHCl<sub>3</sub>. Spar. sol. cold H<sub>2</sub>O. *k* = 0.56 × 10<sup>-14</sup> at 25°. Electrolytic reduction → *o*-phenylenediamine.

*B.HCl*: plates. Decomp. at 155°. Insol. C<sub>6</sub>H<sub>6</sub>, pet. ether.

*B.HI*: plates. Decomp. at 141°.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: needles. M.p. 144°.

*B<sub>2</sub>C<sub>6</sub>H<sub>3</sub>(NO<sub>2</sub>)<sub>3</sub>* - 1 : 3 : 5 : brownish-yellow needles. M.p. 91°.

*Picrate*: red cryst. from EtOH. M.p. 73°.

*N-Me*: see *o*-Nitro-*N*-methylaniline.

*N-Di-Me*: see *o*-Nitro-*N*-dimethylaniline.

*N-Di-Et*: see *o*-Nitro-*N*-diethylaniline.

*N-Dipropyl*: see *o*-Nitro-*N*-dipropylaniline.

*N-Phenyl*: see 2-Nitrodiphenylamine.

*N-Diphenyl*: see 2-Nitrotriphenylamine.

*N-Benzyl*: reddish-yellow prisms from EtOH. M.p. 74–5°.

*N-Dibenzyl*: see *o*-Nitro-*N*-dibenzylaniline.

*N-Formyl*: *o*-nitroformanilide. Yellow needles from EtOH. M.p. 122°. Very sol. AcOH, C<sub>6</sub>H<sub>6</sub>, Me<sub>2</sub>CO, CHCl<sub>3</sub>. Sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, CS<sub>2</sub>.

*N-Acetyl*: see *o*-Nitroacetanilide.

*N-Diacetyl*: cryst. from EtOH. M.p. 94°. Mod. sol. EtOH.

*N-Chloroacetyl*: light yellow needles from EtOH. M.p. 90–3° (88°). Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH. Insol. H<sub>2</sub>O. Alc. KOH → red col.

*N-Dichloroacetyl*: light yellow cryst. from EtOH. M.p. 78–80°. Alc. KOH → orange-red col.

*N-Trichloroacetyl*: yellow needles from EtOH. M.p. 70–2° (65°). Very sol. EtOH. Insol. H<sub>2</sub>O.

*N-Propionyl*: yellow cryst. from EtOH. M.p. 63°. Sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*N-Benzoyl*: golden-yellow needles from EtOH. M.p. 98°. Very sol. Et<sub>2</sub>O. Sol. EtOH. Spar. sol. H<sub>2</sub>O.

*N-Dibenzoyl*: plates from EtOH. M.p. 182°.

Votoček, Burda, *Ber.*, 1915, 48, 1004.

Sakellarios, Jatriles, *Ber.*, 1925, 58, 2286.

Ehrenfeld, Puterbaugh, *Organic Syntheses*, Collective Vol. I, 381.

### *m*-Nitroaniline.

Yellow needles from H<sub>2</sub>O. M.p. 114°. B.p. above 285°. D<sub>4</sub> 1.430. 0.114 parts sol. in 100 parts H<sub>2</sub>O at 20°. Sol. MeOH, Et<sub>2</sub>O. Mod. sol. C<sub>6</sub>H<sub>6</sub>. *k* = 4.0 × 10<sup>-12</sup> at 25°.

*B.HF*: cryst. M.p. 207–9°.

*B.HCl*: cryst. Very sol. H<sub>2</sub>O, EtOH.

*B.HBr*: yellow plates.

*Picrate*: yellow needles from EtOH. M.p. 143°.

*Styphnate*: yellowish needles from C<sub>6</sub>H<sub>6</sub>. M.p. 137°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOEt. Decomp. by CHCl<sub>3</sub>, CCl<sub>4</sub>.

*Dioxalate*: needles from H<sub>2</sub>O. M.p. 119°.

*m*-Nitrobenzoic acid *add. comp.*: yellow needles from Et<sub>2</sub>O. M.p. 88–9°.

2 : 4 : 6-Trinitrobenzoic acid *add. comp.*: yellow needles from hot EtOH. M.p. 139°.

$B, C_6H_3(NO_2)_3-1:3:5$ : yellow needles. M.p. 98°.

*Trichloroacetic acid add. comp.*: pale yellow needles. M.p. 147°.

*N-Me*: see *m*-Nitro-*N*-methylaniline.

*N-Di-Me*: see *m*-Nitro-*N*-dimethylaniline.

*N-Et*: see *m*-Nitro-*N*-ethylaniline.

*N-Di-Et*: see *m*-Nitro-*N*-diethylaniline.

*N-Phenyl*: see 3-Nitrodiphenylamine.

*N-Benzyl*: yellow leaflets from EtOH.Aq. M.p. 107°.

*N-Dibenzyl*: see *m*-Nitro-*N*-dibenzylaniline.

*N-Diphenyl*: see 3-Nitrotriphenylamine.

*N-Formyl*: *m*-nitroformanilide. Cryst. M.p. 134°. Sol. hot  $H_2O$ , EtOH,  $C_6H_6$ . Spar. sol.  $Et_2O$ , ligroin.

*N-Acetyl*: see *m*-Nitroacetanilide.

*N-Diacetyl*: cryst. from  $Et_2O$ . M.p. 76–7°. Sol.  $Et_2O$ ,  $C_6H_6$ .

*N-Chloroacetyl*: plates or prisms from EtOH.Aq. M.p. 116° (101–2°).

*N-Benzoyl*: plates from amyl alcohol. M.p. 157°. Sol.  $Et_2O$ ,  $CHCl_3$ . Spar. sol. cold EtOH. Insol.  $H_2O$ .

*N-Dibenzoyl*: prisms from EtOH. M.p. 150–1°.

Lyford, U.S.P., 1,878,950, (*Chem. Abstracts*, 1933, 27, 307).

Vorontzov, *Chem. Abstracts*, 1931, 25, 4861.

### p-Nitroaniline.

Pale yellow needles from  $H_2O$ . M.p. 148°.  $D_4^{20}$  1.424. Sol. 45 parts boiling, 1250 parts  $H_2O$  at 18.5°. Sol. MeOH, EtOH,  $Et_2O$ . Mod. sol.  $C_6H_6$ , toluene. Non-volatile in steam.  $k = 1.1 \times 10^{-12}$  at 25°.

*B, HF*: m.p. 173–4°.

*B, HCl*: leaflets.

*Picrate*: yellowish-red needles. M.p. 100°.

*N-Me*: see *p*-Nitro-*N*-methylaniline.

*N-Di-Me*: see *p*-Nitro-*N*-dimethylaniline.

*N-Et*: see *p*-Nitro-*N*-ethylaniline.

*N-Di-Et*: see *p*-Nitro-*N*-diethylaniline.

*N-Propyl*: see *p*-Nitro-*N*-propylaniline.

*N-Dipropyl*: see *p*-Nitro-*N*-dipropylaniline.

*N-Phenyl*: see 4-Nitrodiphenylamine.

*N-Diphenyl*: see 4-Nitrotriphenylamine.

*N-Benzyl*: yellow plates from EtOH.Aq. M.p. 147° (142–3°).

*N-Dibenzyl*: see *p*-Nitro-*N*-dibenzylaniline.

*N-Formyl*: *p*-nitroformanilide. Brownish-yellow cryst. from  $H_2O$ . M.p. 194–5°.

*N-Acetyl*: see *p*-Nitroacetanilide.

*N-Chloroacetyl*: leaflets from EtOH. M.p. 185–185.5°.

*N-Dichloroacetyl*: cryst. from toluene. M.p. 128–30°.

*N-Trichloroacetyl*: needles from EtOH. M.p. 146–7°.

*N-Propionyl*: yellowish-brown plates. M.p. 182°.

*N-Benzoyl*: yellowish needles. M.p. 199°. Spar. sol. EtOH. Insol.  $H_2O$ ,  $CHCl_3$ . *N-Formyl*: needles from AcOH. M.p. 165°. Sol. EtOH,  $Me_2CO$ ,  $C_6H_6$ , AcOH, AcOEt. Spar. sol.  $Et_2O$ . Insol.  $H_2O$ .

*N-Dibenzoyl*: prisms from AcOEt. M.p. 203°. Sol.  $Me_2CO$ ,  $C_6H_6$ , AcOH, AcOEt. Insol.  $H_2O$ , EtOH,  $Et_2O$ .

Votoček, Burda, *Ber.*, 1915, 48, 1004.

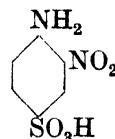
Vasserman, *Chem. Abstracts*, 1931, 25, 5404.

Merrill, U.S.P., 1,786,766, (*Chem. Abstracts*, 1931, 25, 716).

### N-Nitroaniline.

See Phenylnitramine.

### o-Nitroaniline-4-sulphonic Acid (2-Nitrosulphanilic acid)



$C_6H_6O_5N_2S$

MW, 218

Yellow needles. Sol.  $H_2O$ . Less sol. EtOH.

*Chloride*:  $C_6H_5O_4N_2ClS$ . MW, 236.5. Pale yellow plates from  $Et_2O$ . M.p. 59–60°.

*Amide*:  $C_6H_7O_4N_3S$ . MW, 217. Golden plates or needles from  $H_2O$ . M.p. 206–7°.

*N-Phenyl*: see 2-Nitrodiphenylamine-4-sulphonic Acid.

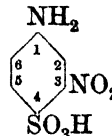
Goslich, *Ann.*, 1876, 180, 103.

Nietzki, Lerch, *Ber.*, 1888, 21, 3220.

Fischer, *Ber.*, 1891, 24, 3788.

Bayer, E.P., 235,598, (*Chem. Abstracts*, 1926, 20, 917).

### m-Nitroaniline-4-sulphonic Acid (3-Nitrosulphanilic acid)



$C_6H_6O_5N_2S$

MW, 218

Needles. Spar. sol. hot  $H_2O$ .

Nietzki, Helbach, D.R.P., 86,096; *Ber.*, 1896, 29, 2448.

Hunter, Sprung, *J. Am. Chem. Soc.*, 1931, 53, 1440.

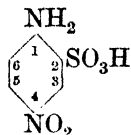


**m-Nitroaniline-6-sulphonic Acid** (5-Nitro-orthanilic acid).

Yellowish-brown prisms.  $k = 8.5 \times 10^{-3}$  at  $25^\circ$ .

Bayer, D.R.P., 294,547, (*Chem. Zentr.*, 1916, II, 780).

**p-Nitroaniline-2-sulphonic Acid** (4-Nitro-orthanilic acid)



$C_6H_6O_5N_2S$  MW, 218

Yellow cryst.

Amide:  $C_6H_7O_4N_3S$ . MW, 217. Yellow needles from  $H_2O$ . M.p.  $207^\circ$ .

N-Phenyl: see 4-Nitrodiphenylamine-2-sulphonic Acid.

Fischer, *Ber.*, 1891, 24, 3789.

Scott, Cohen, *J. Chem. Soc.*, 1922, 121, 2038.

Bayer, E.P., 235,598, (*Chem. Abstracts*, 1926, 20, 917).

**p-Nitroaniline-3-sulphonic Acid** (4-Nitro-metanilic acid).

Pale yellow needles from  $H_2O$ . Sol.  $H_2O$ . Spar. sol. EtOH. Insol. Et<sub>2</sub>O.

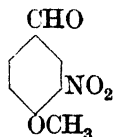
Eger, *Ber.*, 1888, 21, 2581.

Kalle, D.R.P., 150,982, (*Chem. Zentr.*, 1904, I, 1235).

**ω-p-Nitroanilinobenzyl Alcohol.**

See 4-Nitro-N-α-hydroxybenzylaniline.

**3-Nitroanisaldehyde** (3-Nitro-4-methoxybenzaldehyde)



$C_8H_7O_4N$  MW, 181

Needles from  $CHCl_3$ -ligroin or EtOH- $CHCl_3$ . M.p.  $86-7^\circ$ . Sol. usual solvents.

Oxime: anti, needles. M.p.  $170^\circ$ . Very sol. Et<sub>2</sub>O, Me<sub>2</sub>CO. Sol. warm EtOH,  $C_6H_6$ . Syn: yellowish needles. M.p.  $168-70^\circ$ . Spar. sol. usual solvents.

Phenylhydrazone: yellow plates from EtOH. M.p.  $130.5^\circ$ .

p-Nitrophenylhydrazone: red cryst. M.p.  $244^\circ$ .

Einhorn, Grabfield, *Ann.*, 1888, 243, 370.  
Ciusa, *Chem. Zentr.*, 1907, I, 548.

**2-Nitroanisic Acid** (2-Nitro-4-methoxybenzoic acid)



$C_8H_7O_5N$  MW, 197

Needles from  $H_2O$ . M.p.  $195-6^\circ$ . Sol. EtOH, Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O,  $C_6H_6$ .

Ullmann, Dootson, *Ber.*, 1918, 51, 20.

Simonsen, Rau, *J. Chem. Soc.*, 1917, 111, 236.

**3-Nitroanisic Acid** (3-Nitro-4-methoxybenzoic acid).

Needles from  $H_2O$ . M.p.  $186-7^\circ$  ( $195-6^\circ$ ). Sol. EtOH, Et<sub>2</sub>O. Spar. sol. hot  $H_2O$ . Distills under 14 mm. press.

Me ester:  $C_9H_9O_5N$ . MW, 211. Yellow plates. M.p.  $109-10^\circ$ .

Et ester:  $C_{10}H_{11}O_5N$ . MW, 225. Plates from EtOH. M.p.  $97-8^\circ$  ( $71-2^\circ$ ).

Propyl ester:  $C_{11}H_{13}O_5N$ . MW, 239. Cryst. from EtOH. M.p.  $63^\circ$ . B.p.  $213-15^\circ/16$  mm.

Allyl ester:  $C_{11}H_{11}O_5N$ . MW, 237. Cryst. M.p.  $50^\circ$ . B.p.  $207^\circ/11$  mm.

Hexyl ester:  $C_{14}H_{19}O_5N$ . MW, 281. Yellow oil. B.p.  $224-6^\circ/11$  mm.

Chloride:  $C_8H_6O_4NCl$ . MW, 215.5. Cryst. from pet. ether. M.p.  $56^\circ$ .

Nitrile:  $C_8H_6O_3N_2$ . MW, 178. Needles from EtOH. M.p.  $149-50^\circ$ .

Anilide: needles from EtOH. M.p.  $163^\circ$ .

Ashley, Perkin, Robinson, *J. Chem. Soc.*, 1930, 392.

Sabalitschka, Tiedge, *Arch. Pharm.*, 1934, 272, 383.

**3-Nitro-o-anisidine** (3-Nitro-2-aminoanisole)



$C_7H_8O_3N_2$  MW, 168

Yellow or bright scarlet needles from EtOH. M.p.  $76^\circ$ .

N-Me:  $C_8H_{10}O_3N_2$ . MW, 182. Dark red needles. M.p.  $58^\circ$ . Sol. EtOH. N-Acetyl: prisms from Et<sub>2</sub>O. M.p.  $105-6^\circ$ .

N-Acetyl: 3-nitro-o-acetaniside. Pale yellow needles from EtOH.Aq. M.p.  $158-9^\circ$ .

Blanksma, *Chem. Zentr.*, 1908, II, 1826.

Ingold, Ingold, *J. Chem. Soc.*, 1926, 1317, 1324.

**4-Nitro-*o*-anisidine** (4-Nitro-2-amino-anisole).

Orange-red needles from EtOH or Et<sub>2</sub>O. M.p. 118°. Very sol. Me<sub>2</sub>CO. Sol. EtOH, AcOEt, AcOH, boiling C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin.

N-*Me*: orange cryst. from pet. ether. M.p. 87°. N-*Acetyl*: m.p. 126.5–127.5°.

N-*Di-Me*: C<sub>9</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 196. Yellow cryst. from EtOH. M.p. 68–9°.

N-*Acetyl*: 4-nitro-*o*-acetanisidide. Needles from H<sub>2</sub>O. M.p. 175–6°.

N-*Benzoyl*: yellow needles from EtOH. M.p. 160–1°. Spar. sol. EtOH, Et<sub>2</sub>O.

N-*p-Toluenesulphonyl*: cryst. from AcOH. M.p. 128°.

Fabr. de Thann et Mulhouse, D.R.P., 98,637, (*Chem. Zentr.*, 1898, II, 951).

Vermeulen, *Rec. trav. chim.*, 1906, **25**, 18.

Meldola, Woolcott, Wray, *J. Chem. Soc.*, 1896, **69**, 1330.

Ingold, Ingold, *J. Chem. Soc.*, 1926, 1323.

**5-Nitro-*o*-anisidine** (5-Nitro-2-amino-anisole).

Pale yellow needles. M.p. 139–40°.

N-*Me*: yellow needles from MeOH or pet. ether. M.p. 101–2°. N-*Acetyl*: m.p. 119–120.5°.

N-*Di-Me*: yellow needles. M.p. 99°. Sol. boiling EtOH.

N-*Acetyl*: 5-nitro-*o*-acetanisidide. Pale yellow cryst. from AcOEt. M.p. 153–4°. Sol. boiling C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

N-*Benzoyl*: needles from EtOH. M.p. 149–50°.

N-*Benzenesulphonyl*: yellow plates. M.p. 181°.

N-*p-Toluenesulphonyl*: yellow prisms. M.p. 175° (169–70°).

Meldola, Eyre, *Chem. News*, 1901, **83**, 286.

Gribov, Ivanov, Salomatina, *Chem. Abstracts*, 1934, **28**, 1029.

du Pont, U.S.P., 1,963,598, (*Chem. Abstracts*, 1934, **28**, 5084).

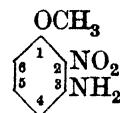
See also previous references.

**6-Nitro-*o*-anisidine** (6-Nitro-2-amino-anisole).

Yellow needles from ligroin. M.p. 67°.

N-*Acetyl*: 6-nitro-*o*-acetanisidide. Pale yellow prisms from MeOH or AcOH.Aq. M.p. 103–4°.

Ingold, Ingold, *J. Chem. Soc.*, 1926, 1318.

**2-Nitro-*m*-anisidine** (2-Nitro-3-amino-anisole)

C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>

MW, 168

Yellow needles. M.p. 143°.

N-*Acetyl*: 2-nitro-*m*-acetanisidide. Brown cryst. M.p. 265°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Sublimes.

Reverdin, Widmer, *Ber.*, 1913, **46**, 4073.

**4-Nitro-*m*-anisidine** (4-Nitro-3-amino-anisole).

Brown needles. M.p. 131°. Sublimes.

N-*Acetyl*: 4-nitro-*m*-acetanisidide. Needles from EtOH. M.p. 125°. Sol. EtOH, AcOH. Mod. sol. ligroin.

Meldola, Stephens, *J. Chem. Soc.*, 1906, **89**, 924.

See also previous reference.

**5-Nitro-*m*-anisidine** (5-Nitro-3-amino-anisole).

Orange cryst. M.p. 120°. Sol. hot H<sub>2</sub>O. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

N-*Acetyl*: 5-nitro-*m*-acetanisidide. Cryst. M.p. 200°. Sol. EtOH, AcOEt. Less sol. C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, ligroin.

Vermeulen, *Rec. trav. chim.*, 1906, **25**, 20.

Höchst, D.R.P., 222,062, (*Chem. Zentr.*, 1910, I, 2001).

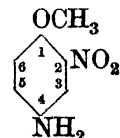
**6-Nitro-*m*-anisidine** (6-Nitro-3-amino-anisole).

Yellow needles from EtOH. M.p. 169° (161°). Sol. EtOH, Me<sub>2</sub>CO, AcOH. Sublimes easily.

N-*Acetyl*: 6-nitro-*m*-acetanisidide. Golden-yellow needles from H<sub>2</sub>O. M.p. 165°. Sol. EtOH, AcOH. Spar. sol. H<sub>2</sub>O. Insol. ligroin.

Reverdin, Widmer, *Ber.*, 1913, **46**, 4072.

Höchst, D.R.P., 285,638, (*Chem. Zentr.*, 1915, II, 511).

**2-Nitro-*p*-anisidine** (2-Nitro-4-amino-anisole)

C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>

MW, 168

Orange prisms and plates from Et<sub>2</sub>O-ligroin. M.p. 57–57.5°. Sol. hot H<sub>2</sub>O, Me<sub>2</sub>CO, EtOH, Et<sub>2</sub>O. Mod. sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. toluene. 10% HCl → pale yellow sol.

$B_2H_2SO_4$ : yellowish-brown cryst. from  $H_2O$ . M.p.  $243^\circ$ . Spar. sol. cold  $H_2O$ .

*N-Di-Me*:  $C_8H_{12}O_3N_2$ . MW, 196. Red cryst. from EtOH. M.p.  $46^\circ$ .

*N-Acetyl*: 2-nitro-*p*-acetanisidide. Yellow needles from  $H_2O$ . M.p.  $153^\circ$ . Sol.  $H_2O$ . Spar. sol.  $C_6H_6$ , ligroin.

*N-Chloroacetyl*: golden-yellow needles from AcOEt. M.p.  $149.5-151.5^\circ$ . Sol.  $Me_2CO$ . Spar. sol.  $CHCl_3$ . Almost insol.  $H_2O$ .

Heidelberger, Jacobs, *J. Am. Chem. Soc.*, 1919, **41**, 1455.

Höchst, D.R.P., 101,778, (*Chem. Zentr.*, 1899, I, 1175).

Klemenc, *Ber.*, 1914, **47**, 1411.

### 3-Nitro-*p*-anisidine (3-Nitro-4-aminoanisole).

Dark red prisms from  $H_2O$  or EtOH. M.p.  $129^\circ$  ( $123^\circ$ ). Sol.  $H_2O$ , EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ . Volatile in steam.

*N-Acetyl*: 3-nitro-*p*-acetanisidide. Yellow needles from EtOH. M.p.  $117^\circ$ . Sol. hot  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ , AcOH.

*N-Benzoyl*: plates from EtOH. M.p.  $140^\circ$ . Sol. hot EtOH,  $C_6H_6$ , AcOH.

*N-o-Nitrobenzoyl*: orange needles. M.p.  $172^\circ$ . Sol.  $Me_2CO$ , hot  $C_6H_6$ . Spar. sol. EtOH, AcOH. Conc.  $H_2SO_4 \rightarrow$  bluish-green col.

*N-m-Nitrobenzoyl*: orange cryst. M.p.  $165-6^\circ$ . Sol.  $Me_2CO$ , EtOH, AcOH.

*N-p-Nitrobenzoyl*: orange needles from AcOH. M.p.  $204^\circ$ . Sol.  $Me_2CO$ , hot AcOH. Spar. sol. EtOH.

Reverdin, *Ber.*, 1896, **29**, 2595; 1911, **44**, 2365.

Reverdin, de Luc, *Ber.*, 1912, **45**, 352.

### *o*-Nitroanisole



$C_7H_7O_3N$

MW, 153

Cryst. M.p.  $10^\circ$ . B.p.  $272^\circ$ ,  $150.5-151^\circ/19$  mm. Insol.  $H_2O$ .  $D_4^{20}$  1.2540.  $n_D^{20}$  1.56204. Volatile in steam.

Ullmann, *Ann.*, 1903, **327**, 114.

Weltz, U.S.P., 1,578,943, (*Chem. Abstracts*, 1926, **20**, 1631).

Clemmensen, U.S.P., 1,875,916, (*Chem. Abstracts*, 1933, **27**, 102).

Aoyama, Morita, *Chem. Abstracts*, 1934, **28**, 141.

### *m*-Nitroanisole.

Needles from EtOH, plates from  $C_6H_6$ -ligroin. M.p.  $38-9^\circ$ . B.p.  $258^\circ$ . Insol.  $H_2O$ .  $D^{18}$  1.373. Volatile in steam.

Bantlin, *Ber.*, 1878, **11**, 2100.

### *p*-Nitroanisole.

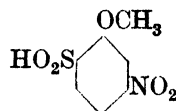
Prisms from EtOH. M.p.  $54^\circ$ . B.p.  $259^\circ$ .  $D_4^{18}$  1.2012. Sol. EtOH,  $Et_2O$ . Sol. 13,923 parts  $H_2O$  at  $15^\circ$ . Spar. sol. cold pet. ether.

v. Blom, *Helv. Chim. Acta*, 1921, **4**, 1029.

v. Erp, *Ber.*, 1923, **56**, 217.

Rarick, Brewster, Dains, *J. Am. Chem. Soc.*, 1933, **55**, 1289.

### *m*-Nitroanisole-6-sulphinic Acid (4-Nitro-2-methoxybenzenesulphinic acid)



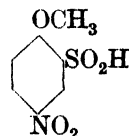
$C_7H_7O_5NS$

MW, 217

Needles from AcOEt. M.p.  $127-9^\circ$ .

Holmes, Ingold, Ingold, *J. Chem. Soc.*, 1926, 1688.

### *p*-Nitroanisole-2-sulphinic Acid (5-Nitro-2-methoxybenzenesulphinic acid)



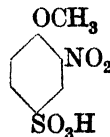
$C_7H_7O_5NS$

MW, 217

Prisms from AcOEt. M.p.  $134-6^\circ$ .

See previous reference.

### *o*-Nitroanisole-4-sulphonic Acid (3-Nitro-4-methoxybenzenesulphonic acid)



$C_7H_7O_6NS$

MW, 233

Plates from  $H_2O$ , prisms from AcOEt- $C_6H_6$ . Very sol.  $H_2O$ , AcOEt.

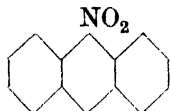
*Me ester*:  $C_8H_9O_6NS$ . MW, 247. Prisms from toluene-ligroin. M.p.  $83^\circ$ . Decomp. by  $H_2O$ .

*Chloride*:  $C_7H_6O_5NSCl$ . MW, 251.5. Needles from  $C_6H_6$ -pet. ether. M.p.  $66^\circ$ . Sol.  $C_6H_6$ , toluene. Spar. sol. ligroin, pet. ether.

*Amide*:  $C_7H_8O_5N_2S$ . MW, 232. Yellowish needles from  $H_2O$ . M.p.  $146.3^\circ$ .

Gnehm, Knecht, *J. prakt. Chem.*, 1906, 74, 92.

### 9-Nitroanthracene (ms-Nitroanthracene)



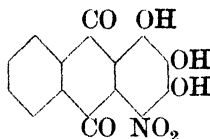
$C_{14}H_9O_2N$

MW, 223

Yellow needles from EtOH, prisms from AcOH or xylene. M.p.  $146^\circ$ . Distills in vacuo above  $300^\circ$ . Very sol.  $C_6H_6$ ,  $CS_2$ . Spar. sol. EtOH, AcOH. Insol. aq. alkalis.

Dimroth, *Ber.*, 1901, 34, 221; D.R.P., 127,399, (*Chem. Zentr.*, 1902, I, 235).

### 4-Nitroanthragallol (4-Nitro-1:2:3-trihydroxyanthraquinone)



$C_{14}H_7O_7N$

MW, 301

Golden-brown cryst. from hot EtOH-ligroin. M.p.  $224^\circ$  decomp. Sol. EtOH, AcOH. Less sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Insol.  $H_2O$ , pet. ether. *Triacetyl*: yellow needles from  $Ac_2O$ -AcOH. M.p.  $233^\circ$ .

*Tribenzoyl*: cryst. from AcOH. M.p.  $209^\circ$ . Sol. EtOH,  $C_6H_6$ , AcOH. Insol.  $H_2O$ .

Bamberger, Bück, *Monatsh.*, 1897, 18, 288; 1901, 22, 719.

### 3-Nitroanthranilic Acid (3-Nitro-o-amino-benzoic acid)



$C_7H_6O_4N_2$

MW, 182

Yellow needles from  $H_2O$ . M.p.  $208-9^\circ$ . Very sol. EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ ,  $CHCl_3$ . D<sup>15</sup> 1.558. Spar. volatile in steam.

*Et ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Yellow plates. M.p.  $109^\circ$ . Mod. sol.  $H_2O$ , EtOH. Readily volatile in steam. *N-Acetyl*: needles from  $H_2O$ . M.p.  $102^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ ,  $CHCl_3$ , pet. ether. Sublimes at  $140^\circ$ . *N-Benzoyl*: cryst. from ligroin. M.p.  $85.5^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. ligroin.

*N-Me*:  $C_8H_8O_4N_2$ . MW, 196. Reddish-brown

needles from EtOH.Aq. M.p.  $146^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

*N-Acetyl*: yellow needles from hot  $H_2O$  or AcOH. M.p.  $180-1^\circ$ .

Chapman, Stephen, *J. Chem. Soc.*, 1925, 127, 1795.

Keller, *Arch. Pharm.*, 1908, 246, 32.

Zacharias, *J. prakt. Chem.*, 1891, 43, 435.

### 4-Nitroanthranilic Acid (4-Nitro-o-amino-benzoic acid).

Orange prisms from EtOH.Aq. M.p.  $269^\circ$ . Sol. xylene. Spar. sol. hot  $H_2O$ . Sweet taste.

*Me ester*:  $C_8H_8O_4N_2$ . MW, 196. Orange needles. M.p.  $157^\circ$ . Sol. EtOH, ligroin.

*Et ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Orange prisms from EtOH. M.p.  $100^\circ$ . Very sol. hot EtOH,  $Et_2O$ . *N-Acetyl*: pale yellow plates. M.p.  $112^\circ$ .

*N-Et*:  $C_9H_{10}O_4N_2$ . MW, 210. Golden-yellow needles or plates. M.p.  $223^\circ$ . *Et ester*:  $C_{11}H_{14}O_4N_2$ . MW, 238. Pale yellow needles from EtOH or  $C_6H_6$ . M.p.  $80^\circ$ .

*N-Phenyl*: see 5-Nitrodiphenylamine-2-carboxylic Acid.

*N-Acetyl*: needles from EtOH.Aq. M.p.  $217^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $C_6H_6$ , ligroin. *Amide*: pale yellow needles from EtOH. M.p.  $218-23^\circ$ . *Guamidinium salt*: cryst. M.p.  $247^\circ$ .

Wheeler, Barnes, *Am. Chem. J.*, 1898, 20, 221.

Wheeler, Jones, *Am. Chem. J.*, 1910, 44, 444.

I.G., E.P., 285,877, (*Chem. Abstracts*, 1929, 23, 156).

See also first reference above.

### 5-Nitroanthranilic Acid (5-Nitro-o-amino-benzoic acid).

Yellow needles from EtOH.Aq. M.p.  $280^\circ$  ( $263^\circ$ ). Sol. hot  $H_2O$ , EtOH,  $Et_2O$ . Insol.  $C_6H_6$ ,  $CHCl_3$ , xylene. Hot conc. KOH  $\rightarrow$  5-nitrosalicylic acid.

*Me ester*:  $C_8H_8O_4N_2$ . MW, 196. Greenish-yellow needles from EtOH.Aq. M.p.  $168^\circ$ . Sol. usual org. solvents. Spar. sol.  $H_2O$ . Spar. volatile in steam. Sublimes.

*Et ester*:  $C_9H_{10}O_4N_2$ . MW, 210. Yellow needles from EtOH. M.p.  $148^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $Me_2CO$ ,  $CHCl_3$ , AcOH. Spar. sol. pet. ether. *N-Acetyl*: needles from EtOH. M.p.  $153^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Insol.  $H_2O$ .

*N-Me*:  $C_8H_8O_4N_2$ . MW, 196. Pale yellow leaflets from EtOH. M.p.  $259^\circ$  decomp. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ . *Et ester*:  $C_{10}H_{12}O_4N_2$ . MW, 224. Greenish-yellow needles from

EtOH.Aq. M.p. 103°. Sol. EtOH, Et<sub>2</sub>O. Sublimes. *Methylamide*: golden-yellow needles from hot H<sub>2</sub>O. M.p. 204°. Sol. EtOH. Spar. sol. hot H<sub>2</sub>O, Et<sub>2</sub>O. Sublimes in needles. *N-Acetyl*: plates from Et<sub>2</sub>O. M.p. 66°. Sol. EtOH. Spar. sol. cold H<sub>2</sub>O.

*N-Phenyl*: see 4-Nitrodiphenylamine-2-carboxylic Acid.

*N-o-Tolyl*: C<sub>11</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub>. MW, 272. Yellow needles from EtOH. M.p. 253-4°.

*N-p-Tolyl*: yellow needles from AcOH. M.p. 262-5°.

*Amide*: C<sub>7</sub>H<sub>7</sub>O<sub>3</sub>N<sub>3</sub>. MW, 181. Yellow needles from EtOH-Me<sub>2</sub>CO. M.p. 230°. Sol. Me<sub>2</sub>CO. Spar. sol. hot H<sub>2</sub>O, EtOH.

*Methylamide*: yellow needles from EtOH.Aq. M.p. 230-1° decomp. Very sol. warm H<sub>2</sub>O, EtOH. Spar. sol. CHCl<sub>3</sub>. Insol. C<sub>6</sub>H<sub>6</sub>.

*Dimethylamide*: m.p. 213-14°.

*Ethylamide*: golden-yellow needles from EtOH. M.p. 156° decomp. Sol. AcOH. Spar. sol. H<sub>2</sub>O, CHCl<sub>3</sub>.

*Anilide*: C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>N<sub>3</sub>. MW, 257. Needles from AcOH. M.p. 203°. Spar. sol. H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Methylanilide*: m.p. 183-4°.

*Ethylanilide*: m.p. 144-5°.

*Ethyl-o-toluidide*: m.p. 147-8°.

*N-Acetyl*: yellow needles from H<sub>2</sub>O. M.p. 216-17°.

*Hydrazide*: yellow needles from H<sub>2</sub>O. M.p. 214-18° decomp. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Reduces NH<sub>3</sub>.AgNO<sub>3</sub> and Fehling's.

Thieme, *J. prakt. Chem.*, 1891, **43**, 470.

Grohmann, *Ber.*, 1891, **24**, 3810.

Kratz, *J. prakt. Chem.*, 1896, **53**, 215.

Bayer, D.R.P., 309,951, (*Chem. Zentr.*, 1919, II, 179).

Chapman, Stephen, *J. Chem. Soc.*, 1925, **127**, 1796.

**6-Nitroanthranilic Acid** (6-Nitro-o-amino-benzoic acid).

Yellow needles from H<sub>2</sub>O. M.p. 184°. Very sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Sol. hot H<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, CS<sub>2</sub>. Sweet taste. Dil. H<sub>2</sub>SO<sub>4</sub> → *m*-nitroaniline.

*Anilide*: C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>N<sub>3</sub>. MW, 257. Yellow needles from CHCl<sub>3</sub>. M.p. 137°.

*N-Carbethoxyl*: C<sub>10</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 254. Yellowish needles from EtOH.Aq. or H<sub>2</sub>O. M.p. 187°.

*N-Acetyl*: cryst. from H<sub>2</sub>O. M.p. 212-14° decomp. Sol. hot EtOH, cold Me<sub>2</sub>CO, hot AcOEt. Spar. sol. hot CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. Et<sub>2</sub>O, pet. ether. *Amide*: cryst. M.p. 218-19°.

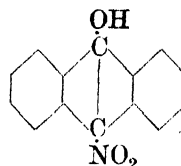
*N-Propionyl*: cryst. from AcOEt. M.p. 218°.

Curtius, Semper, *Ber.*, 1913, **46**, 1168.

Bogert, Chambers, *J. Am. Chem. Soc.*, 1905, **27**, 653.

Bogert, Seil, *ibid.*, 1309.

**ms-Nitroanthranol** (9-Nitro-10-hydroxy-anthracene)



C<sub>14</sub>H<sub>9</sub>O<sub>3</sub>N MW, 239

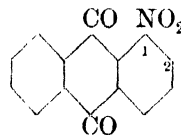
Red needles. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.

*Acetyl*: yellow plates or needles from CHCl<sub>3</sub>-pet. ether. M.p. 182°.

*Benzoyl*: yellow prisms from CHCl<sub>3</sub>-pet. ether. M.p. 238° decomp. Spar. sol. EtOH.

Meyer, Sander, *Ann.*, 1913, **396**, 150.

**1-Nitroanthraquinone**



C<sub>14</sub>H<sub>7</sub>O<sub>4</sub>N MW, 253

Needles from AcOH. M.p. 230°. B.p. 270°/7 mm. Sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOH. Spar. sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Sublimes in yellow plates.

Boettger, Petersen, *Ann.*, 1873, **166**, 147.

Lauth, *Bull. soc. chim.*, 1903, **29**, 1133.

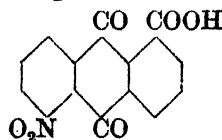
**2-Nitroanthraquinone.**

Yellow needles from EtOH or AcOH. M.p. 184.5-185°. Distills in vacuo. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, hot AcOH, amyl alcohol. Mod. sol. EtOH, Et<sub>2</sub>O. Spar. sol. ligroin. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. → reddish-yellow on heating.

Scholl, Schneider, Eberle, *Ber.*, 1904, **37**, 4434.

Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2048.

**5-Nitroanthraquinone-1-carboxylic Acid**



C<sub>15</sub>H<sub>7</sub>O<sub>6</sub>N MW, 297

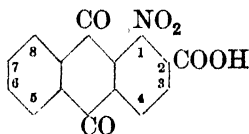
Yellow plates. Decomp. above 330°. Sol. boiling Me<sub>2</sub>CO, Py, PhNO<sub>2</sub>. Spar. sol. boiling

EtOH, AcOH, toluene. Insol. Et<sub>2</sub>O, ligroin. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

*Nitrile*: C<sub>15</sub>H<sub>6</sub>O<sub>4</sub>N<sub>2</sub>. MW, 278. Yellow plates. M.p. about 390°. Sol. boiling Py, PhNO<sub>2</sub>. Spar. sol. AcOH, toluene. Insol. EtOH, Et<sub>2</sub>O.

Ullmann, v. der Schalk, *Ann.*, 1912, **388**, 208.

### 1-Nitroanthraquinone-2-carboxylic Acid



C<sub>15</sub>H<sub>7</sub>O<sub>6</sub>N MW, 297

Needles from EtOH or AcOH. M.p. 285-7°. Sol. hot Me<sub>2</sub>CO. Insol. Et<sub>2</sub>O, ligroin.

*Me ester*: C<sub>16</sub>H<sub>9</sub>O<sub>6</sub>N. MW, 311. M.p. 249-51°.

*Et ester*: C<sub>17</sub>H<sub>11</sub>O<sub>6</sub>N. MW, 325. Pale yellow plates from AcOH. M.p. 233-4°.

*Propyl ester*: C<sub>18</sub>H<sub>13</sub>O<sub>6</sub>N. MW, 339. M.p. 182-3°.

*Isopropyl ester*: m.p. 204-6°.

*Butyl ester*: C<sub>19</sub>H<sub>15</sub>O<sub>6</sub>N. MW, 353. M.p. 4-5°.

17sec.-n-*Butyl ester*: m.p. 155-6°.

*Isobutyl ester*: m.p. 159-60°.

*Amyl ester*: C<sub>20</sub>H<sub>17</sub>O<sub>6</sub>N. MW, 367. M.p. 166-8°.

*Isoamyl ester*: m.p. 165-7°.

*Hexyl ester*: C<sub>21</sub>H<sub>19</sub>O<sub>6</sub>N. MW, 381. M.p. 153-4°.

*Cetyl ester*: m.p. 105-6°.

*Allyl ester*: C<sub>18</sub>H<sub>11</sub>O<sub>6</sub>N. MW, 337. M.p. 184-5°.

*Benzyl ester*: m.p. 211-12°.

*Menthyl ester*: m.p. 189-90°.

*Amide*: C<sub>15</sub>H<sub>8</sub>O<sub>5</sub>N<sub>2</sub>. MW, 296. Yellow plates or prisms from AcOH. M.p. 299-301°. Spar. sol. most org. solvents.

Sah, Ma, *J. Chinese Chem. Soc.*, 1933, **1**, 51.

Badische, D.R.P., 256,344, (*Chem. Zentr.*, 1913, **I**, 759).

Terres, *Ber.*, 1913, **46**, 1639.

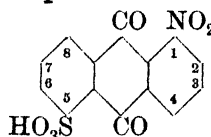
### 5-Nitroanthraquinone-2-carboxylic Acid.

Yellow needles from AcOH. Sol. AcOH, chlorobenzene, hot toluene.

*Amide*: C<sub>15</sub>H<sub>8</sub>O<sub>5</sub>N<sub>2</sub>. MW, 296. Cryst. from AcOH or PhNO<sub>2</sub>. M.p. 330° decomp.

Eckert, *Monatsh.*, 1914, **35**, 295.

### 1-Nitroanthraquinone-5-sulphonic Acid



C<sub>14</sub>H<sub>7</sub>O<sub>7</sub>NS

MW, 333

Cryst. from H<sub>2</sub>O.

*Chloride*: C<sub>14</sub>H<sub>6</sub>O<sub>6</sub>NCIS. MW, 351.5. Yellow needles from PhNO<sub>2</sub>. M.p. 277°. Sol. hot C<sub>6</sub>H<sub>6</sub>, toluene. Spar. sol. Me<sub>2</sub>CO, AcOH.

Ullmann, Kertész, *Ber.*, 1919, **52**, 552.

Maki, Nagai, *Chem. Abstracts*, 1931, **25**, 948.

### 1-Nitroanthraquinone-6-sulphonic Acid (5-Nitroanthraquinone-2-sulphonic acid).

Yellowish plates from dil. HNO<sub>3</sub>. M.p. 255° decomp. Spar. sol. cold H<sub>2</sub>O.

*Chloride*: C<sub>14</sub>H<sub>6</sub>O<sub>6</sub>NCIS. MW, 351.5. Yellowish needles. M.p. 194°. Sol. hot toluene, AcOH. Insol. EtOH, Et<sub>2</sub>O.

Claus, *Ber.*, 1882, **15**, 1514.

Höchst, D.R.P., 145,188, (*Chem. Zentr.*, 1903, **II**, 1038).

### 1-Nitroanthraquinone-7-sulphonic Acid (8-Nitroanthraquinone-2-sulphonic acid).

Cryst. powder. M.p. 250° decomp. Sol. H<sub>2</sub>O, EtOH, AcOH. Alkalis → red col.

See last reference above and also

Claus, *Ber.*, 1882, **15**, 1516.

### 1-Nitroanthraquinone-8-sulphonic Acid.

Cryst. from H<sub>2</sub>O.

*Chloride*: yellow needles. M.p. 245°.

Ullmann, Kertész, *Ber.*, 1919, **52**, 552.

Maki, Nagai, *Chem. Abstracts*, 1931, **25**, 948.

### 2-Nitroanthraquinone-7-sulphonic Acid (7-Nitroanthraquinone-2-sulphonic acid).

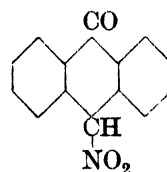
Cryst. from H<sub>2</sub>O.

*Na salt*: white needles from H<sub>2</sub>O.

Gubelmann, Weiland, Stallmann, *J. Am. Chem. Soc.*, 1931, **53**, 1035.

Newport Chemical Corp., U.S.P., 1,810,010, (*Chem. Abstracts*, 1931, **25**, 4559).

### ms-Nitroanthrone



C<sub>14</sub>H<sub>9</sub>O<sub>3</sub>N

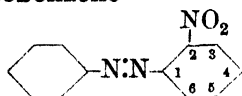
MW, 239

Cryst. from  $C_6H_6$ -ligroin. M.p.  $140^\circ$ . Alkalis  $\rightarrow$  red sols.

*Di-Me acetal*: needles. M.p.  $135^\circ$  decomp. Very sol.  $CS_2$ . Spar. sol. EtOH,  $C_6H_6$ ,  $CHCl_3$ .

Meyer, *Organic Syntheses*, Collective Vol. I, 382.

### 2-Nitroazobenzene



$C_{12}H_9O_2N_3$

MW, 227

Dark orange-red prisms from EtOH. M.p.  $70.5-71^\circ$ . Sol. EtOH,  $CHCl_3$ , AcOH,  $C_6H_6$ , ligroin.

Bamberger, Hübner, *Ber.*, 1903, 36, 3818.

### 3-Nitroazobenzene.

Orange needles. M.p.  $95.5-96^\circ$ . Sol. EtOH, AcOH, ligroin.

Bamberger, Hübner, *Ber.*, 1903, 36, 3811.

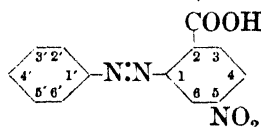
### 4-Nitroazobenzene.

Orange plates or needles from ligroin. M.p.  $135^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ , ligroin.

See previous reference and also

Angeli, Alessandri, *Atti accad. Lincei*, 1911, 20, 171.

### 5-Nitroazobenzene-2-carboxylic Acid (o-Benzeneazo-p-nitrobenzoic acid)



$C_{13}H_9O_4N_3$

MW, 271

*Anilide*:  $C_{19}H_{14}O_3N_4$ . MW, 332. Orange-red cryst. from EtOH. M.p.  $180.5^\circ$ .

Sachs, Sichel, *Ber.*, 1903, 36, 4375.

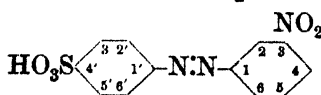
### 2-Nitroazobenzene-4-carboxylic Acid (p-Benzeneazo-m-nitrobenzoic acid).

Red needles from EtOH. M.p.  $215^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Et ester*:  $C_{15}H_{13}O_4N_3$ . MW, 299. Red needles. M.p.  $139^\circ$ .

Werner, Peters, *Ber.*, 1906, 39, 191.

### 3-Nitroazobenzene-4'-sulphonic Acid



$C_{12}H_9O_5N_3S$

MW, 307

Leaflets. Sol.  $H_2O$ . Reduction  $\rightarrow$  m-phenylenediamine + sulphanilic acid.

*K salt*: orange-red plates. Spar. sol. cold  $H_2O$ .

Janovsky, *Monatsh.*, 1882, 3, 504.

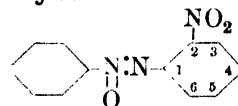
### 4-Nitroazobenzene-4'-sulphonic Acid.

Orange-yellow needles +  $3H_2O$  from  $H_2O$ , plates from dil.  $HNO_3$ . 3.1 parts sol. 100 parts  $H_2O$  at  $10^\circ$ .  $Zn + HCl \rightarrow$  p-phenylenediamine + sulphanilic acid.

*K salt*: orange-yellow plates. 0.161 parts sol. 100 parts  $H_2O$  at  $17^\circ$ , 1.76 parts sol. 100 parts  $H_2O$  at  $82^\circ$ .

Janovsky, *Monatsh.*, 1883, 4, 276.

### 2-Nitroazoxybenzene



$C_{12}H_9O_3N_3$

MW, 243

Yellow needles or prisms. M.p.  $49^\circ$ . Very sol.  $Et_2O$ ,  $C_6H_6$ . Less sol. EtOH.

Angeli, Alessandri, *Atti. accad. Lincei*, 1911, 20, 896.

Zinin, *Ann.*, 1860, 114, 218.

### 4-Nitroazoxybenzene.

Pale yellow cryst. from EtOH. M.p.  $153^\circ$ .

See previous references.

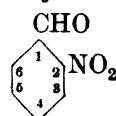
### Nitrobarbituric Acid.

See Dilituric Acid.

### Nitrobenzaldazine.

See under Nitrobenzaldehyde.

### o-Nitrobenzaldehyde



$C_7H_5O_3N$

MW, 151

Bright yellow needles from  $H_2O$ . M.p.  $43-4^\circ$ . B.p.  $153^\circ/23$  mm. Sol. most org. solvents. Spar. sol.  $H_2O$ . Volatile in steam. Ox.  $\rightarrow$  o-nitrobenzoic acid. Exposure of  $C_6H_6$  sol. to sunlight  $\rightarrow$  o-nitrosobenzoic acid.

*syn-Oxime*: cryst. from  $C_6H_6$ . M.p.  $154^\circ$ . Boiling the  $C_6H_6$  sol.  $\rightarrow$  anti-oxime.

*anti-Oxime*: needles. M.p.  $102-3^\circ$  ( $96-7^\circ$ ). Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $CS_2$ , pet. ether. Sol. alkalis. Irradiation of  $C_6H_6$  sol.  $\rightarrow$  syn-oxime.

*Semicarbazone*: yellow needles from  $H_2O$ . M.p.  $256^\circ$  ( $242^\circ$ ) decomp. Spar. sol.  $H_2O$ . Insol. most org. solvents.

*Hydrazone*: see *o*-Nitrobenzylidenehydrazine.  
*o*-Nitrophenylhydrazone: orange cryst. from Py. M.p. 223°.

*o*-Tolylhydrazone: scarlet prisms. M.p. 149.5°.  
*m*-Tolylhydrazone: red prisms. M.p. 129.5°.  
*p*-Tolylhydrazone: red prisms. M.p. 150.5°.

*Benzylidenehydrazone*: *o*-nitrobenzaldazine. Yellow cryst. M.p. 105°.

*Di-Me acetal*: C<sub>9</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 197. B.p. 274-6°, 138-9°/11 mm.

*Di-Et acetal*: C<sub>11</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 225. B.p. 154-6°/18 mm., 148°/11 mm.

*Diacetate*: see *o*-Nitrobenzylidene diacetate.

*Anil*: see *o*-Nitrobenzylideneaniline.

2-Chloroanil: m.p. 116.5°.

3-Chloroanil: m.p. 77-8°.

4-Chloroanil: m.p. 92.5°.

2-Bromoanil: m.p. 119°.

3-Bromoanil: m.p. 77-8°.

4-Bromoanil: m.p. 99°.

Bamberger, Fodor, *Ber.*, 1910, **43**, 3334.

Einhorn, *Ber.*, 1884, **17**, 121.

Ciamician, Silber, *Ber.*, 1901, **34**, 2041; 1903, **36**, 4268.

Fischer, Giehe, *Ber.*, 1897, **30**, 3058.

Curtius, Lublin, *Ber.*, 1900, **33**, 2463.

Brady, Dunn, *J. Chem. Soc.*, 1913, **103**, 1614.

Senier, Clarke, *J. Chem. Soc.*, 1914, **105**, 1920.

Chattaway, Clemo, *J. Chem. Soc.*, 1923, **123**, 3041.

**m-Nitrobenzaldehyde.**

Needles from H<sub>2</sub>O. M.p. 58°. B.p. 164°/23 mm. Sol. most org. solvents. Spar. sol. H<sub>2</sub>O. Heat of comb. C<sub>p</sub> 800.3 Cal. Volatile in steam. Ox. → *m*-nitrobenzoic acid.

*syn*-Oxime: plates from Et<sub>2</sub>O. Heated above 85° → *anti*-oxime. *Me ether*: needles from Et<sub>2</sub>O. M.p. 72-4°.

*anti*-Oxime: needles from Et<sub>2</sub>O. M.p. 121-3°. D<sub>120</sub> 1.043. Irradiation in C<sub>6</sub>H<sub>6</sub> → *syn*-oxime. *Me ether*: needles. M.p. 63-63.5°.

*Semicarbazone*: yellow needles from EtOH. M.p. 246°.

*Thiosemicarbazone*: needles from EtOH. M.p. 163°.

*Phenylsemicarbazone*: m.p. 195°.

*Phenylthiosemicarbazone*: needles from EtOH. M.p. 132°.

*Hydrazone*: see *m*-Nitrobenzylidenehydrazine.

*o*-Tolylhydrazone: reddish-orange prisms. M.p. 170°.

*m*-Tolylhydrazone: yellow prisms. M.p. 127.5°.

*p*-Tolylhydrazone: yellow cryst. M.p. 150.5°.

*Benzylidenehydrazone*: *m*-nitrobenzaldazine. Yellow plates from CHCl<sub>3</sub>. M.p. 125°.

*p*-Bromophenylhydrazone: m.p. 151°.

*Di-Me acetal*: b.p. 162-4°/19 mm. D<sub>15</sub> 1.209.

*Di-Et acetal*: b.p. 178°/21 mm. D<sub>15</sub> 1.131.

*Diacetate*: see *m*-Nitrobenzylidene diacetate.

*Anil*: see *m*-Nitrobenzylideneaniline.

4-Chloroanil: m.p. 81°. *B, HCl*: m.p. 132°.

4-Nitroanil: m.p. 153°.

Ehrlich, *Ber.*, 1882, **15**, 2010.

Freundler, *Compt. rend.*, 1904, **138**, 289.

Goldschmidt, *Ber.*, 1890, **23**, 2170; 1891, **24**, 2809; 1904, **37**, 183.

Brady, Dunn, *J. Chem. Soc.*, 1913, **103**, 1614.

Curtius, Lublin, *Ber.*, 1900, **33**, 2463.

Chattaway, Clemo, *J. Chem. Soc.*, 1923, **123**, 3041.

**p-Nitrobenzaldehyde.**

Prisms from H<sub>2</sub>O. M.p. 106°. Sublimes. Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, pet. ether. Spar. volatile in steam. Mod. stable towards oxidising agents. Used in preparation of triphenylmethane dyes.

*syn*-Oxime: plates from amyl alcohol. M.p. 182-4°. Fusion → *anti*-oxime. *Me ether*: needles. M.p. 67-8°. Volatile in steam. *Et ether*: m.p. 70-1°.

*anti*-Oxime: needles from H<sub>2</sub>O. M.p. 133° (128-9°). Sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, pet. ether. Irradiation in C<sub>6</sub>H<sub>6</sub> → *syn*-oxime. *Me ether*: needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 105° (101-2°). Volatile in steam. *Et ether*: m.p. 107-8°.

*Semicarbazone*: needles + 2H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 211°.

*Hydrazone*: see *p*-Nitrobenzylidenehydrazine.

*Phenylhydrazone*: dark red cryst. with metallic lustre from EtOH. M.p. 153-4°.

*p*-Nitrophenylhydrazone: cryst. M.p. 249°.

2:4-Dinitrophenylhydrazone: orange needles from xylene or quinoline. M.p. 320°.

*o*-Tolylhydrazone: m.p. 162°.

*m*-Tolylhydrazone: crimson needles. M.p. 109°.

*p*-Tolylhydrazone: m.p. 161.5°.

*Benzylidenehydrazone*: *p*-nitrobenzaldazine. Yellow needles from AcOH. M.p. 300°.

*Di-Me acetal*: m.p. 23-5°. B.p. 294-6°.

*Diacetate*: see *p*-Nitrobenzylidene diacetate.

*Anil*: see *p*-Nitrobenzylideneaniline.

2-Chloroanil: m.p. 121°.

3-Chloroanil: m.p. 77-8°.



4-Chloroanil : m.p. 128°.

Fischer, Grieff, *Ber.*, 1880, **13**, 670; 1881, **14**, 2525.

Law, Perkin, *J. Chem. Soc.*, 1908, **98**, 1635.

Fischer, Giebe, *Ber.*, 1897, **30**, 3057.

Thiele, Winter, *Ann.*, 1900, **311**, 355.

Ciamician, Silber, *Ber.*, 1903, **36**, 4269.

Goldschmidt, Kjellin, *Ber.*, 1891, **24**, 2553.

Brady, Dunn, *J. Chem. Soc.*, 1913, **103**, 1614.

Curtius, Lublin, *Ber.*, 1900, **33**, 2463.

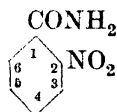
Brady, *J. Chem. Soc.*, 1931, 758.

Chattaway, Clemo, *J. Chem. Soc.*, 1923, **123**, 3041.

ω-Nitrobenzaldoxime.

See Benzonitrolic Acid.

o-Nitrobenzamide



$C_7H_6O_3N_2$  MW, 166

Needles from EtOH.Aq. M.p. 174-6°. B.p. 317°. Sol. EtOH, hot  $H_2O$ .

N-Di-Me :  $C_9H_{10}O_3N_2$ . MW, 194. Cryst. from EtOH. M.p. 78°.

Reissert, *Ber.*, 1908, **41**, 3815.

Kalle, D.R.P., 204,477, (*Chem. Zentr.*, 1909, I, 114).

Löb, *Ber.*, 1894, **27**, 3093.

m-Nitrobenzamide.

Needles from  $H_2O$ . M.p. 141-3°. B.p. 310-15°.

N-Me :  $C_8H_8O_3N_2$ . MW, 180. M.p. 174°.

N-Et :  $C_9H_{10}O_3N_2$ . MW, 194. M.p. 120°. Sol. hot  $H_2O$ .

N-Acetyl : m.p. 198°. Sol. hot  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $Me_2CO$ .

N-Benzoyl : m.p. 133-4°.

Claus, *J. prakt. Chem.*, 1895, **51**, 401.

Pinner, *Ber.*, 1895, **28**, 483.

Kao, Ma, *J. Chem. Soc.*, 1931, 443.

p-Nitrobenzamide.

Needles from  $H_2O$ . M.p. 200° (197-8°).

N-Me : cryst. from  $H_2O$ . M.p. 218°.

N-Et : cryst. from EtOH. M.p. 149°.

Basterfield, Greig, *Chem. Zentr.*, 1933, II, 1022.

Reichenbach, Beilstein, *Ann.*, 1864, **132**, 143.

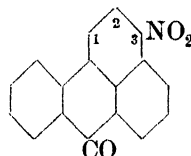
Blanksma, *Rec. trav. chim.*, 1902, **21**, 417.

See also last reference above.

Nitrobenzanilide.

See under Nitrobenzoic Acid.

3-Nitrobenzanthrone



$C_{17}H_9O_3N$

MW, 275

Greenish-yellow needles from AcOH. M.p. 244-5°. Sol. conc.  $H_2SO_4$  → yellow sol. Red. → 3-aminobenzanthrone.

Lüttringhaus, Neresheimer, *Ann.*, 1929, **473**, 285.

Nitrobenzene

$C_6H_5 \cdot NO_2$

MW, 123

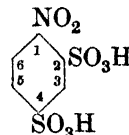
$C_6H_5O_2N$

Liq. with characteristic odour. F.p. 5.85°. B.p. 210-85°/760 mm., 184.5°/400 mm., 139.9°/100 mm., 120.2°/50 mm., 108.2°/30 mm., 85.4°/10 mm., 53.1°/1 mm.  $D_4^{20}$  1.3440 (solid),  $D_4^{20}$  1.2229 (liq.),  $D_4^{10}$  1.2125,  $D_4^{25}$  1.1732.  $n_D^{20}$  1.55296. Misc. with most org. solvents. Spar. sol.  $H_2O$ . Aq. sol. tastes sweet. Volatile in steam. Vapour is poisonous. Heat of comb.  $C_r$  734.8 Cal. Alkaline or acid reduction → nitrosobenzene → β-phenylhydroxylamine → aniline. Electrolytic reduction in weak acid → aniline; in strong acid → p-aminophenol.

Nitrobenzeneazophthol.

See under Naphthoquinone.

Nitrobenzene-2 : 4-disulphonic Acid



$C_6H_5O_8NS_2$

MW, 283

Hygrosopic needles from  $H_2O$ . M.p. 199° decomp.

Heinzelmann, *Ann.*, 1877, **188**, 162.

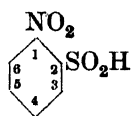
Nitrobenzene-3 : 5-disulphonic Acid.

Dichloride :  $C_6H_3O_6NCl_2S_2$ . MW, 320. Cryst. from  $CCl_4$ . M.p. 97-8°.

Diamide :  $C_6H_7O_6N_3S_2$ . MW, 281. Leaflets from  $H_2O$ . M.p. 242°.

Heinzelmann, *Ann.*, 1877, **188**, 165.

Bennet, Willis, *J. Chem. Soc.*, 1929, 266.

Nitrobenzene-*o*-sulphinic Acid

$C_6H_5O_4NS$  MW, 187

Needles. M.p.  $134^\circ$  ( $124^\circ$ ). Sol. EtOH, AcOH. Spar. sol.  $H_2O$ .

*Na salt*: yellow leaflets. M.p.  $123^\circ$ .

*Me ester*:  $C_7H_5O_4NS$ . MW, 201. Plates from MeOH. M.p.  $106^\circ$ .

*Et ester*:  $C_8H_9O_4NS$ . MW, 215. Prisms from EtOH. M.p.  $58^\circ$ .

Claasz, *Ann.*, 1911, **380**, 314.

Zincke, Farr, *Ann.*, 1912, **391**, 73.

Nitrobenzene-*m*-sulphinic Acid.

Needles. M.p.  $98^\circ$  ( $95-6^\circ$ ). Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $Me_2CO$ . Insol.  $C_6H_6$ . Phenol + conc.  $H_2SO_4 \rightarrow$  blue col.

*Et ester*: prisms. M.p.  $100^\circ$ .

*Bromide*:  $C_6H_4O_3NBrS$ . MW, 250. M.p.  $68^\circ$ .

*Anilide*: m.p.  $122^\circ$ .

Limpricht, *Ann.*, 1894, **278**, 242.

Flürscheim, *J. prakt. Chem.*, 1905, **71**, 526.

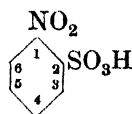
Nitrobenzene-*p*-sulphinic Acid.

Needles or prisms from  $H_2O$ . M.p.  $159^\circ$  ( $120^\circ$ ). Sol. AcOH, EtOH. Spar. sol.  $H_2O$ .

*Me ester*: m.p.  $141^\circ$ .

Zincke, Lenhardt, *Ann.*, 1913, **400**, 16.

Limpricht, *Ber.*, 1887, **20**, 1240.

Nitrobenzene-*o*-sulphonic Acid

$C_6H_5O_5NS$  MW, 203

Very hygroscopic cryst. M.p. about  $70^\circ$ .

*Chloride*:  $C_6H_4O_4NCIS$ . MW, 221.5. Cryst. from ligroin. M.p.  $68-9^\circ$ . Sol.  $Et_2O$ . Spar. sol. ligroin.

*Bromide*:  $C_6H_4O_4NBrS$ . MW, 266. M.p.  $63-4^\circ$ .

*Amide*:  $C_6H_5O_4N_2S$ . MW, 202. Needles from 50% EtOH. M.p.  $193^\circ$ .

*Me-amide*:  $C_7H_8O_4N_2S$ . MW, 216. M.p.  $113^\circ$ .

*Et-amide*:  $C_8H_{10}O_4N_2S$ . MW, 230. M.p.  $103^\circ$ .

*Hydrazide*:  $C_6H_7O_4N_3S$ . MW, 217. Prisms. M.p.  $101^\circ$  decomp.

*Anilide*:  $C_{13}H_{10}O_4N_2S$ . MW, 278. Cryst. from  $Me_2CO$ . M.p.  $115^\circ$ .

Dict. of Org. Comp.—III.

*N-Me-anilide*:  $C_{13}H_{12}O_4N_2S$ . MW, 292. M.p.  $73^\circ$ .

*$\beta$ -Naphthalide*:  $C_{16}H_{12}O_4N_2S$ . MW, 238. Needles from EtOH. M.p.  $138^\circ$ .

Obermiller, *J. prakt. Chem.*, 1914, **89**, 70, 82.

Ullmann, Gross, *Ber.*, 1910, **43**, 2703.

Davies, Storrie, Tucker, *J. Chem. Soc.*, 1931, 624.

Nitrobenzene-*m*-sulphonic Acid.

Deliquescent plates. Sol. hot EtOH.  $SOCl_2$  at  $180-200^\circ \rightarrow$  *m*-dichlorobenzene.

*p-Nitrophenyl ester*: m.p.  $132-3^\circ$ .

*Chloride*: needles from ligroin. M.p.  $64^\circ$  ( $61^\circ$ ). Sol. hot EtOH. Insol.  $H_2O$ .

*Bromide*: prisms from  $C_6H_6$ -ligroin. M.p.  $68^\circ$ .

*Amide*: needles from 50% EtOH. M.p.  $167-8^\circ$ .

*Me amide*: yellow prisms from EtOH. M.p.  $125^\circ$ .

*Et-amide*: plates from EtOH. M.p.  $81^\circ$ .

*N-Dichloroamide*:  $C_6H_4O_4N_2Cl_2S$ . MW, 271. Yellow plates from  $CHCl_3$ -pet. ether. M.p.  $121^\circ$ .

*Hydrazide*: prisms. M.p.  $130^\circ$  decomp.

*Anilide*: yellow plates from EtOH. M.p.  $126^\circ$ .

*Benzylamide*:  $C_{13}H_{12}O_4N_2S$ . MW, 292. Yellow plates from EtOH. M.p.  $101^\circ$ .

*o-Toluidide*:  $C_{13}H_{12}O_4N_2S$ . MW, 292. Yellow plates from EtOH. M.p.  $164^\circ$ .

*p-Toluidide*: yellow plates from EtOH. M.p.  $132^\circ$ .

Obermiller, *J. prakt. Chem.*, 1914, **89**, 70, 81.

Nitrobenzene-*p*-sulphonic Acid.

Hygroscopic cryst. M.p.  $95^\circ$ .

*Et ester*:  $C_8H_9O_5NS$ . MW, 231. Plates. M.p.  $91^\circ$ .

*Phenyl ester*:  $C_{12}H_9O_5NS$ . MW, 279. Needles from AcOH. M.p.  $114^\circ$ .

*m-Nitrophenyl ester*: m.p.  $133^\circ$ .

*p-Nitrophenyl ester*: m.p.  $156^\circ$ .

*p-Tolyl ester*:  $C_{13}H_{11}O_5NS$ . MW, 293. M.p.  $106^\circ$ .

*Chloride*: needles from ligroin. M.p.  $80^\circ$ .

*Amide*: prisms from 50% EtOH. M.p.  $179-80^\circ$ .

*Me-amide*: m.p.  $110^\circ$ .

*Et-amide*: m.p.  $103^\circ$ .

*Hydrazide*: yellow prisms. M.p.  $150-2^\circ$  decomp.

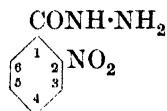
*Anilide*: cryst. from EtOH. M.p.  $135-6^\circ$ .

*N-Me-anilide*: m.p.  $117-18^\circ$ .

p-Toluidide : m.p. 179–80°.

Obermiller, *J. prakt. Chem.*, 1914, **89**, 84.  
Demény, *Rec. trav. chim.*, 1929, **48**, 1145.  
Bell, *J. Chem. Soc.*, 1928, 2776.

**o-Nitrobenzhydrazide** (o-Nitrobenzoylhydrazine)



$C_7H_7O_3N_3$

MW, 181

Yellowish-brown cryst. from  $H_2O$ . M.p. 123°. Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

Curtius, Trachmann, *J. prakt. Chem.*, 1895, **51**, 168.

**m-Nitrobenzhydrazide** (m-Nitrobenzoylhydrazine).

Needles from  $H_2O$ . M.p. 152°. Spar. sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .

Meng, Sah, *Science Reports National Tsinghua Univ.*, 1934, **2**, 348.

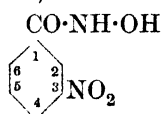
See also previous reference.

**p-Nitrobenzhydrazide** (p-Nitrobenzoylhydrazine).

Yellowish needles from  $H_2O$ . M.p. 210°. Spar. sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .

Curtius, Trachmann, *J. prakt. Chem.*, 1895, **51**, 168.

**m-Nitrobenzhydroxamic Acid** (N-m-Nitrobenzoyl-hydroxylamine)



$C_7H_6O_4N_2$

MW, 182

Granules from  $CHCl_3$  or  $C_6H_6$ . M.p. 153° (151°). Gives red col. with  $FeCl_3$ .Aq.

O-Benzoyl : needles from MeOH. M.p. 153–4°.

O-m-Nitrobenzoyl : needles from EtOH. M.p. 153–4°.

Werner, Skiba, *Ber.*, 1899, **32**, 1662.

Meisenheimer, Patzig, *Ber.*, 1906, **39**, 2542.

Angelico, Fanara, *Gazz. chim. ital.*, 1901, **31**, 33.

**p-Nitrobenzhydroxamic Acid.**

Needles from EtOH or xylene. M.p. 177° decomp. Sol. hot  $H_2O$ , NaOH.Aq.

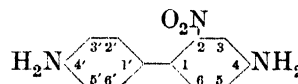
O-Benzoyl : plates from AcOH or EtOH. M.p. 185° decomp. (178°).

O-p-Nitrobenzoyl : needles from EtOH. M.p. 174° decomp. (173–6°).

Werner, Skiba, *Ber.*, 1899, **32**, 1665.

Holleman, *Rec. trav. chim.*, 1897, **16**, 187.

**2-Nitrobenzidine** (2-Nitro-4 : 4'-diaminodiphenyl)



$C_{12}H_{11}O_2N_3$

MW, 229

Red needles from  $H_2O$ . M.p. 143° (140–1°). A labile form, m.p. 117°, is also described.

4'-N-Acetyl : yellow plates from EtOH or  $H_2O$ . M.p. 186–7°.

4 : 4'-N : N-Diacetyl : cryst. from AcOH. Does not melt below 310°. Insol.  $H_2O$ , EtOH,  $Et_2O$ , AcOEt,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. AcOH.

4 : 4'-N : N-Di-p-toluenesulphonyl : m.p. 164°.

4 : 4'-N : N-Dibenzylidene : brownish-yellow cryst. from  $C_6H_6$ . M.p. 157°.

4 : 4'-N : N-Di-p-nitrobenzylidene : yellow cryst. from xylene. M.p. 205–6°.

Cain, May, *J. Chem. Soc.*, 1910, **97**, 725.

Ponte, *Chem. Abstracts*, 1934, **28**, 2345.

Täuber, *Ber.*, 1890, **23**, 796.

Sako, *Bull. Chem. Soc. Japan*, 1934, **9**, 150.

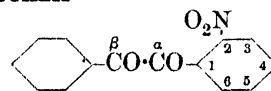
**3-Nitrobenzidine.**

Red cryst. from EtOH. M.p. 208–10°. Prac. insol. EtOH.

3 : 4'-N : N-Diacetyl : yellow needles from dil. AcOH. M.p. 249–50°.

Le Fèvre, Turner, *J. Chem. Soc.*, 1928, 253.

**2-Nitrobenzil**



$C_{14}H_9O_4N$

MW, 255

Yellow needles with green reflex from EtOH. M.p. 102° (98°). Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. EtOH, AcOH.  $CrO_3 \rightarrow$  benzoic and o-nitrobenzoic acids.

Monoxime :  $\alpha$ -, needles or plates from EtOH. M.p. 185° decomp.  $\beta$ -, m.p. 265° decomp.

Dioxime : yellowish prisms from EtOH. M.p. 244° decomp. Prac. insol. EtOH. Sol. alkalis with red col.

List, *Ber.*, 1893, **26**, 2453.

Chattaway, Coulson, *J. Chem. Soc.*, 1928, 1083.

**3-Nitrobenzil.**

Cryst. from EtOH or Me<sub>2</sub>CO. M.p. 120°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, hot AcOH. Mod. sol. EtOH. CrO<sub>3</sub> → benzoic and m-nitrobenzoic acids.

See last reference above.

**4-Nitrobenzil.**

Yellow plates or needles from EtOH. M.p. 142°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Sn + HCl → 4'-aminodeoxybenzoin. CrO<sub>3</sub> → benzoic and p-nitrobenzoic acids.

*Dioxime*: two forms. (i) M.p. 225° decomp. Prac. insol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. EtOH at 170° → (ii). (ii) Needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 185°. Sol. EtOH.

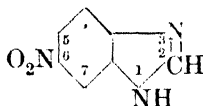
*Phenylhydrazine*: (i) yellow plates. M.p. 200°. (ii) Orange plates. M.p. 162°.

*Phenylosazone*: orange needles from AcOH. M.p. 216°.

List, *Ber.*, 1893, **26**, 2456.

Hausmann, *Ber.*, 1890, **23**, 532.

Chattaway, Coulson, *J. Chem. Soc.*, 1928, 1083.

**6-Nitrobenzimidazole**

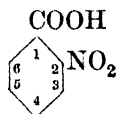
C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>N<sub>3</sub> MW, 163

Needles from H<sub>2</sub>O. M.p. 203°. Sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Sol. acids and alkalis.

N-Me: C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub>. MW, 177. *Methiodide*: yellow prisms and needles from H<sub>2</sub>O. M.p. 259°.

Bamberger, Berlé, *Ann.*, 1893, **273**, 340.

Fischer, Hess, *Ber.*, 1903, **36**, 3968.

**o-Nitrobenzoic Acid**

C<sub>7</sub>H<sub>5</sub>O<sub>4</sub>N MW, 167

Needles from H<sub>2</sub>O. M.p. 146-8°. D<sub>4</sub><sup>20</sup> 1.575. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Spar. volatile in steam. Does not melt under boiling H<sub>2</sub>O. Heat of comb. C<sub>p</sub> 730.4 Cal., C<sub>v</sub> 731.1 Cal.  $k = 6.56 \times 10^{-3}$  at 25°,  $1.6 \times 10^{-3}$  at 100°. NaHg → azobenzene-2:2'-dicarboxylic acid. NaOH + Zn dust → hydrazobenzene-2:2'-dicarboxylic acid. Zn + HCl → anthranilic acid.

*Me ester*: C<sub>8</sub>H<sub>7</sub>O<sub>4</sub>N. MW, 181. M.p. -13°. B.p. 275°, 183°/22 mm., 169°/19 mm. D<sub>20</sub>

1.2855. Sol. MeOH, EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

*Et ester*: C<sub>9</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 195. M.p. 30°. B.p. 173°/18 mm.

*Propyl ester*: C<sub>10</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 209. B.p. 173-5°/16 mm.

*d-Amyl ester*: C<sub>12</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 237. B.p. 238°/69 mm. D<sub>18</sub> 1.135.  $n_D^{20}$  1.5132.  $[\alpha]_D^{18} + 1.17^\circ$ .

*l-Menthyl ester*: C<sub>17</sub>H<sub>23</sub>O<sub>4</sub>N. MW, 305. Prisms from EtOH. M.p. 62-4°. B.p. 185°/2 mm. D<sub>4</sub><sup>20</sup> 1.1140, D<sub>180</sub> 0.983.  $[\alpha]_D^{65} - 125^\circ$ .

*Anhydride*: C<sub>14</sub>H<sub>8</sub>O<sub>7</sub>N<sub>2</sub>. MW, 316. M.p. 135°. Explodes on rapid heating. Sol. EtOH, AcOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O.

*Chloride*: see o-Nitrobenzoyl chloride.

*Amide*: see o-Nitrobenzamide.

*Nitrile*: see o-Nitrobenzonitrile.

*Anilide*: o-nitrobenzanilide. C<sub>13</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 242. Needles from EtOH. M.p. 155°.

N-Me-anilide: C<sub>14</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 256. Needles from EtOH. M.p. 94°.

2:4-Dichloroanilide: C<sub>13</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>Cl<sub>2</sub>. MW, 311. Plates from EtOH. M.p. 153°.

o-Nitroanilide: C<sub>13</sub>H<sub>9</sub>O<sub>3</sub>N<sub>3</sub>. MW, 287. Needles from AcOH. M.p. 167-8°.

*Hydrazide*: see o-Nitrobenzhydrazide.

*Azide*: C<sub>7</sub>H<sub>4</sub>O<sub>3</sub>N<sub>4</sub>. MW, 192. Yellow prisms from Et<sub>2</sub>O. M.p. 37.5° (36°).

Beilstein, Kuhlberg, *Ann.*, 1872, **163**, 138.

Reimer, Gatewood, *J. Am. Chem. Soc.*, 1920, **42**, 1475.

Noyes, *Ber.*, 1883, **16**, 53.

Monnet, Reverdin, Noeltig, *Ber.*, 1879, **12**, 443.

Curtius, Melsbach, *J. prakt. Chem.*, 1910, **81**, 523.

Adams, Wirth, French, *J. Am. Chem. Soc.*, 1918, **40**, 425.

Pinnow, Müller, *Ber.*, 1895, **28**, 151.

**m-Nitrobenzoic Acid.**

Prisms. M.p. 140-1°. Known in two labile and one stable modifications. Melts under hot H<sub>2</sub>O. D<sub>4</sub><sup>20</sup> 1.494. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O, pet. ether. Heat of comb. C<sub>p</sub> 727 Cal., C<sub>v</sub> 727.7 Cal.  $k = 3.45 \times 10^{-4}$  at 25°. Zn + HCl → m-aminobenzoic acid. Zn + NaOH → azoxybenzene-3:3'-dicarboxylic acid and azobenzene-3:3'-dicarboxylic acid.

*Me ester*: needles. M.p. 78°. B.p. 279°. Spar. sol. EtOH.

*Et ester*: m.p. 47° (41°). B.p. 296-8°.

*d-Amyl ester*: b.p. 223-5°/52 mm. D<sub>19</sub> 1.144.  $n_D^{20}$  1.5187.  $[\alpha]_D^{18} + 5.58^\circ$ .

*l-Menthyl ester*: b.p. 186°/2 mm. D<sub>4</sub><sup>20</sup> 1.1173. D<sub>180</sub> 0.9787.  $[\alpha]_D^{20} - 82.52^\circ$ .

*Anhydride*: cryst. from  $C_6H_6$ . M.p. 163°. Insol. EtOH,  $Et_2O$ .

*Chloride*: see m-Nitrobenzoyl chloride.

*Amide*: see m-Nitrobenzamide.

*Nitrile*: see m-Nitrobenzonitrile.

*Anilide*: m-nitrobenzanilide. Leaflets from EtOH. M.p. 153–4°.

2:4-Dichloroanilide: plates from AcOH. M.p. 183°.

o-Nitroanilide: m.p. 138°.

m-Nitroanilide: m.p. 227°.

p-Nitroanilide: m.p. 249°.

p-Toluidide: needles from EtOH. M.p. 162°.

*Hydrazide*: see m-Nitrobenzhydrazide.

*Azide*: plates from dil. EtOH. M.p. 68°.

Kamm, Segur, *Organic Syntheses*, Collective Vol. I, 383.

Müller, *Z. physik. Chem.*, 1912, **79**, 172.

Marshall, Acree, *Ber.*, 1910, **43**, 2329.

Adams, Wirth, French, *J. Am. Chem. Soc.*, 1918, **40**, 425.

Beilstein, Kuhlberg, *Ann.*, 1867, **143**, 336.

### p-Nitrobenzoic Acid.

Leaflets from  $H_2O$ . M.p. 238°. Sublimes.  $D^{20}_D$  1.61. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $Me_2CO$ , boiling  $H_2O$ . Spar. sol.  $CS_2$ ,  $C_6H_6$ . Insol. ligroin. Heat of comb.  $C_p$  728.8 Cal.,  $C_r$  729.6 Cal.  $k = 4.0\text{--}4.3 \times 10^{-4}$  at 25°.  $(NH_4)_2S \rightarrow$  p-aminobenzoic acid.  $NaHg \rightarrow$  azoxybenzene-4:4'-dicarboxylic acid. Unaffected by Zn + HCl.

*Me ester*: m.p. 96°.

*Et ester*: leaflets from EtOH. M.p. 57°.

*Propyl ester*: leaflets from EtOH. M.p. 35°.

*Isopropyl ester*: needles from EtOH. M.p. 108–10°.

d-Amyl ester: b.p. 250–2°/80 mm.  $D^{17}_D$  1.14.  $n_D$  1.5203.  $[\alpha]^{25}_D + 6.29^\circ$ .

l-Menthyl ester: prisms from EtOH. M.p. 61–3°.  $D^{25}_D$  1.077.  $[\alpha]^{25}_D - 88.3^\circ$ .

*Benzyl ester*:  $C_{14}H_{11}O_4N$ . MW, 257. Yellow plates from EtOH. M.p. 84°.

2-Naphthyl ester:  $C_{17}H_{12}O_4N$ . MW, 294. Needles from MeOH. M.p. 166°.

*Anhydride*: yellowish leaflets from AcOEt. M.p. 189–90°.

*Chloride*: see p-Nitrobenzoyl chloride.

*Amide*: see p-Nitrobenzamide.

*Nitrile*: see p-Nitrobenzonitrile.

*Anilide*: p-nitrobenzanilide. Leaflets from  $Et_2O$ . M.p. 211° (204°).

o-Nitroanilide: leaflets from AcOH. M.p. 216°.

m-Nitroanilide: plates from EtOH. M.p. 185°.

p-Nitroanilide: m.p. 266°.

p-Toluidide: needles from EtOH. M.p. 203° (192°).

*Hydrazide*: see p-Nitrobenzhydrazide.

*Azide*: plates from dil. EtOH. M.p. 69°.

Kamm, Matthews, *Organic Syntheses*, Collective Vol. I, 385.

Robertson, *ibid.*, 389.

Schlosser, Skraup, *Monatsh.*, 1881, **2**, 519.

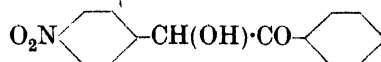
Beilstein, Kuhlberg, *Ann.*, 1872, **163**, 128.

Meyer, *Monatsh.*, 1901, **22**, 426.

### Nitrobenzoic Acid Sulphonic Acid.

See Nitrosulphobenzoic Acid.

### 4'-Nitrobenzoin



$C_{14}H_{11}O_4N$

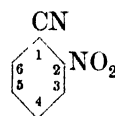
MW, 257

*Acetyl*: leaflets from 90% EtOH. M.p. 125°. Sol.  $CHCl_3$ ,  $Me_2CO$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ , pet. ether.

*Benzoyl*: cryst. M.p. 137°.

Francis, Keane, *J. Chem. Soc.*, 1911, **99**, 346.

### o-Nitrobenzonitrile



$C_7H_4O_2N_2$

MW, 148

Needles from  $H_2O$  or AcOH. M.p. 109–10°. Sol. EtOH,  $CHCl_3$ , AcOH,  $CS_2$ ,  $C_6H_6$ , hot  $H_2O$ . Spar. sol. pet. ether.  $Zn + HCl \rightarrow$  o-aminobenzonitrile + o-aminobenzamide.

Pinnow, Müller, *Ber.*, 1895, **28**, 151.

Reissert, *Ber.*, 1908, **41**, 3812.

Kalle, D.R.P., 210,563, (*Chem. Zentr.*, 1909, II, 78).

### m-Nitrobenzonitrile.

Needles from  $H_2O$ . M.p. 117–18°. Sol.  $Et_2O$ , AcOH. Mod. sol. EtOH, hot  $H_2O$ . Insol. pet. ether. Sublimes. Volatile in steam.

Beilstein, Kuhlberg, *Ann.*, 1868, **146**, 336.

Sandmeyer, *Ber.*, 1885, **18**, 1494.

### p-Nitrobenzonitrile.

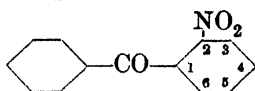
Leaflets from EtOH. M.p. 149°. Sol.  $CHCl_3$ , AcOH, hot EtOH. Spar. sol.  $H_2O$ ,  $Et_2O$ . Sublimes. Volatile in steam.

Bogert, Kohnstamm, *J. Am. Chem. Soc.*, 1903, **25**, 479.

Borsche, *Ber.*, 1909, **42**, 3597.

Neber, Hartung, Ruópp, *Ber.*, 1925, **58**, 1239.

## 2-Nitrobenzophenone

 $C_{13}H_9O_3N$ 

MW, 227

Cryst. from EtOH. M.p.  $105^\circ$ . Mod. sol. EtOH.  $Sn + HCl \rightarrow$  2-aminobenzophenone.  $Sn + AcOH \rightarrow$  phenylanthranil.  $HNO_3 \rightarrow$  2:2', 2:3' and 2:4'-dinitrobenzophenone.

*Oxime*: needles from dil. EtOH.

v. Tatschalow, *J. prakt. Chem.*, 1902, **65**, 308.

Geigy, Koenigs, *Ber.*, 1885, **18**, 2403.

Meyer, *Ber.*, 1893, **26**, 1251.

Boëtius, Römisch, *Ber.*, 1935, **68**, 1927.

## 3-Nitrobenzophenone.

Needles from EtOH. M.p.  $95^\circ$  ( $92^\circ$ ). B.p.  $234^\circ/18$  mm.  $SnCl_2 + HCl \rightarrow$  3-aminobenzophenone.  $Zn + NaOH \rightarrow$  3:3'-dibenzoylazoxybenzene.  $HNO_3 \rightarrow$  2:3', 3:3' and 3:4'-dinitrobenzophenone.

*Phenylhydrazone*: yellow needles from EtOH. M.p.  $116^\circ$ .

Becker, *Ber.*, 1882, **15**, 2092.

Geigy, Koenigs, *Ber.*, 1885, **18**, 2401.

Montagne, *Rec. trav. chim.*, 1916, **36**, 260.

## 4-Nitrobenzophenone.

Plates from EtOH. M.p.  $138^\circ$ . Sol. hot EtOH. Mod. sol.  $C_6H_6$ . Spar. sol.  $CS_2$ , ligroin.  $Sn + HCl \rightarrow$  4-aminobenzophenone.  $Zn + NaOH \rightarrow$  4:4'-dibenzoylazobenzene and 4:4'-dibenzoylazoxybenzene.  $HNO_3 \rightarrow$  2:4', 3:4' and 4:4'-dinitrobenzophenone.

*Monoxime*:  $\alpha$ -, yellow needles from  $C_6H_6$ . M.p.  $159^\circ$ . *Me ether*: yellow leaflets from MeOH. M.p.  $93^\circ$ . *N-Me*: yellow plates from MeOH. M.p.  $147^\circ$ .  $\beta$ -, cryst. from  $C_6H_6$ . M.p.  $136^\circ$ . *Me ether*: plates from MeOH. M.p.  $96^\circ$ . *N-Me*: yellow needles from EtOH. M.p.  $186^\circ$ .

*Phenylhydrazone*: orange-red cryst. from EtOH. M.p.  $142^\circ$ .

Baeyer, Villiger, *Ber.*, 1904, **37**, 605.

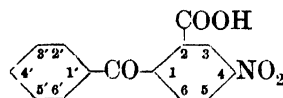
Carré, *Ann. chim. phys.*, 1910, **19**, 228.

Schreeter, *Ber.*, 1909, **42**, 3360 (*Foot-note*).

Reddellien, *J. prakt. Chem.*, 1915, **91**, 237.

Sutton, Taylor, *J. Chem. Soc.*, 1931, 2192.

Brady, Mehta, *J. Chem. Soc.*, 1924, **125**, 2297.

4-Nitrobenzophenone-2-carboxylic Acid  
(5-Nitro-o-benzoylbenzoic acid) $C_{14}H_9O_5N$ 

MW, 271

Leaflets + 1MeOH from MeOH. M.p.  $212^\circ$ . Spar. sol.  $H_2O$ , MeOH,  $Et_2O$ . Conc.  $H_2SO_4 \rightarrow$  2-nitroanthraquinone.

*Me ester*:  $C_{15}H_{11}O_5N$ . MW, 285. Prisms from MeOH. M.p.  $124^\circ$ .

Rainer, *Monatsh.*, 1908, **29**, 178, 431.

5-Nitrobenzophenone-2-carboxylic Acid  
(4-Nitro-o-benzoylbenzoic acid).

Pale yellow needles from  $H_2O$  or  $C_6H_6$ . M.p.  $164-5^\circ$  ( $161^\circ$ ). Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ , AcOH. Spar. sol.  $H_2O$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  2-nitroanthraquinone.

*Me ester*: prisms from MeOH- $Et_2O$ . M.p.  $104-5^\circ$ . Sol. MeOH,  $Et_2O$ ,  $CHCl_3$ .

See previous reference and also

Kliegl, *Ber.*, 1905, **38**, 295.

3'-Nitrobenzophenone-2-carboxylic Acid  
(2-o-Nitrobenzoylbenzoic acid).

Pale yellow prisms from MeOH. M.p.  $186^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ .

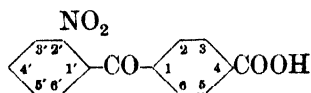
*Me ester*: prisms from propyl alcohol. M.p.  $108-9^\circ$  ( $105^\circ$ ). Sol. EtOH,  $C_6H_6$ . Spar. sol.  $H_2O$ .

*Et ester*:  $C_{16}H_{13}O_5N$ . MW, 299. M.p.  $84^\circ$ .

Lang, *Monatsh.*, 1905, **26**, 972.

Rainer, *Monatsh.*, 1908, **29**, 180.

Hahn, Reid, *J. Am. Chem. Soc.*, 1924, **46**, 1651.

2'-Nitrobenzophenone-4-carboxylic Acid  
(4-o-Nitrobenzoylbenzoic acid) $C_{14}H_9O_5N$ 

MW, 271

Cryst. from EtOH. M.p.  $235-6^\circ$ . Sol. EtOH,  $Me_2CO$ , AcOH. Spar. sol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol. ligroin.

Kliegl, *Ber.*, 1908, **41**, 1849.

3'-Nitrobenzophenone-4-carboxylic Acid  
(4-m-Nitrobenzoylbenzoic acid).

Plates from EtOH. M.p.  $242^\circ$ . Sol. AcOH. Spar. sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

*Chloride*:  $C_{14}H_8O_4NCl$ . MW, 289.5. Cryst.

from  $C_6H_6$ . M.p.  $94.5^\circ$ . Sol. AcOH,  $CHCl_3$ ,  $C_6H_6$ .

Amide:  $C_{14}H_{10}O_4N_2$ . MW, 270. Plates from EtOH.Aq. M.p.  $204^\circ$ .

Limpricht, Lenz, *Ann.*, 1895, **286**, 316.

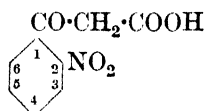
**4'-Nitrobenzophenone-4-carboxylic Acid** (4-p-Nitrobenzoylbenzoic acid).

Needles. M.p.  $255^\circ$ . Sol. EtOH,  $CHCl_3$ , AcOH. Spar. sol.  $C_6H_6$ , ligroin.

Chloride: cryst. from  $CS_2$ . M.p.  $124^\circ$ .

Limpricht, Samietz, *Ann.*, 1895, **286**, 330.

**o-Nitrobenzoylformic Acid**



$C_9H_7O_5N$

MW, 209

Needles from  $H_2O$ . M.p.  $117-20^\circ$  decomp. Sol. EtOH. Spar. sol.  $C_6H_6$ . Insol. pet. ether. Boiling  $H_2O \rightarrow$  o-nitroacetophenone. Mild red.  $\rightarrow$  indigo.

Et ester:  $C_{11}H_{11}O_5N$ . MW, 237. Needles from EtOH. M.p.  $35-6^\circ$ .

Needham, Perkin, *J. Chem. Soc.*, 1904, **85**, 152.

Gabriel, Gerhard, *Ber.*, 1921, **54**, 1069.

**m-Nitrobenzoylformic Acid.**

Cryst. from  $H_2O$ . M.p.  $150^\circ$  decomp.

Et ester: prisms from EtOH. M.p.  $78-9^\circ$ .

Bülow, Hailer, *Ber.*, 1902, **35**, 933.

Reich, *Chem. Abstracts*, 1918, **12**, 1877.

**p-Nitrobenzoylformic Acid.**

Needles from  $C_6H_6$ . M.p.  $135^\circ$  decomp. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ , pet. ether. Spar. sol.  $C_6H_6$ . Alc.  $FeCl_3 \rightarrow$  reddish-brown col. Heated with alkalis or dil.  $H_2SO_4 \rightarrow$  p-nitroacetophenone.

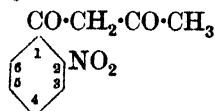
Me ester:  $C_{10}H_9O_5N$ . MW, 223. Prisms. M.p.  $106-7^\circ$ . Sol.  $CHCl_3$ ,  $C_6H_6$ , pet. ether. Spar. sol. EtOH. Alc.  $FeCl_3 \rightarrow$  violet-brown col.

Et ester: needles from EtOH. M.p.  $74-6^\circ$ . Sol. EtOH,  $Et_2O$ .  $FeCl_3 \rightarrow$  violet-brown col.

Perkin, Bellenot, *J. Chem. Soc.*, 1886, **49**, 443.

Bülow, Hailer, *Ber.*, 1902, **35**, 931.

**o-Nitrobenzoylacetone**



$C_{10}H_9O_4N$

MW, 207

Yellow cryst. from ligroin. M.p.  $55^\circ$ . Sol. EtOH,  $Et_2O$ , alkalis. Spar. sol. ligroin. Prac. insol.  $H_2O$ .

Gevekoht, *Ann.*, 1883, **221**, 332.

**m-Nitrobenzoylacetone.**

Needles from EtOH. M.p.  $114-15^\circ$ .  $SnCl_2 \rightarrow$  m-aminobenzoylacetone.

Gabriel, Gerhard, *Ber.*, 1921, **54**, 1617.

**p-Nitrobenzoylacetone.**

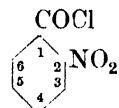
Needles. M.p.  $102^\circ$  (softens at  $98^\circ$ ).  $SnCl_2 \rightarrow$  p-aminobenzoylacetone.

Gabriel, Gerhard, *Ber.*, 1921, **54**, 1618.

**Nitrobenzoylbenzoic Acid.**

See Nitrobenzophenone-carboxylic Acid.

**o-Nitrobenzoyl chloride**



$C_7H_4O_3NCl$

MW, 185.5

M.p.  $20^\circ$ . B.p.  $148^\circ/9$  mm.,  $105^\circ/0.5$  mm. ( $97^\circ/1$  mm.).

Meyer, *Monatsh.*, 1901, **22**, 426.

Schroeter, Eisleb, *Ann.*, 1909, **367**, 128.

**m-Nitrobenzoyl chloride.**

Cryst. M.p.  $35^\circ$ . B.p.  $275-8^\circ$ ,  $183-4^\circ/50-5$  mm.,  $154-5^\circ/18$  mm. Very sol.  $Et_2O$ .

See first reference above.

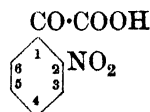
**p-Nitrobenzoyl chloride.**

Yellow needles from ligroin. M.p.  $75^\circ$ . B.p.  $202-5^\circ/105$  mm.,  $150-2^\circ/15$  mm.

Meyer, *Monatsh.*, 1901, **22**, 426.

Adams, Jenkins, *Organic Syntheses*, Collective Vol. I, 387.

**o-Nitrobenzoylformic Acid** (o-Nitrophenylglyoxylic acid)



$C_8H_5O_5N$

MW, 195

Prisms +  $H_2O$  from  $H_2O$ . M.p.  $46-7^\circ$ , anhyd.  $123^\circ$  ( $156-7^\circ$  decomp.). Sol.  $Me_2CO$ , AcOH. Mod. sol. EtOH. Spar. sol.  $Et_2O$ , AcOEt, ligroin, toluene.  $FeSO_4 + NaOH \rightarrow$  isatin.

Et ester:  $C_{10}H_9O_5N$ . MW, 223. Cryst. from  $C_6H_6$ -ligroin. M.p.  $43-44.5^\circ$ . Sol. most org. solvents. Oxime: needles from boiling  $H_2O$ , leaflets from EtOH.Aq. M.p.  $163^\circ$ . Sol. warm EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ ,  $CS_2$ .

**Amide**:  $C_8H_6O_4N_2$ . MW, 194. Prisms from  $H_2O$ . M.p.  $199^\circ$ . Sol. boiling  $H_2O$ . Spar. sol. EtOH,  $Et_2O$ .

**Nitrile**:  $C_8H_4O_3N_2$ . MW, 176. Prisms from pet. ether. M.p.  $54^\circ$ .

**Oxime**: brownish-yellow cryst. +  $H_2O$  from EtOH.Aq. M.p.  $87-8^\circ$ .

Borsche, *Ber.*, 1909, **42**, 3599.

Claissen, Shadwell, *Ber.*, 1879, **12**, 352.

Claissen, Thompson, *ibid.*, 1945 (Note).

Heller, *Ber.*, 1911, **44**, 2419.

**m-Nitrobenzoylformic Acid** (m-Nitro-phenylglyoxylic acid).

Prisms from  $C_6H_6$ . M.p.  $105^\circ$ .

Baker, *J. Chem. Soc.*, 1931, 2422.

**p-Nitrobenzoylformic Acid** (p-Nitrophenylglyoxylic acid).

Cryst. from  $C_6H_6$ . M.p.  $150^\circ$ .

See previous reference.

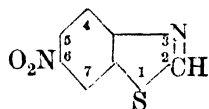
**Nitrobenzoylhydrazine**.

See Nitrobenzhydrazide.

**Nitrobenzoylpyridine**.

See Nitrophenyl pyridyl Ketone.

**6-Nitrobenzthiazole** (5-Nitrobenzthiazole)



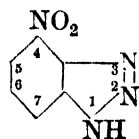
$C_7H_4O_2N_2S$

MW, 180

Yellowish leaflets. M.p.  $176-7^\circ$ .

Jacobson, Kwaysser, *Ann.*, 1893, **277**, 244.

**4-Nitro-1 : 2 : 3-benztriazole** (7-Nitro-1 : 2 : 3-benztriazole)



$C_8H_4O_2N_4$

MW, 164

Pale yellow needles from EtOH. M.p.  $229^\circ$ .

Fries, Güterbock, Kühn, *Ann.*, 1934, **511**, 229.

Borsche, Rantscheff, *Ann.*, 1911, **379**, 164.

**5-Nitro-1 : 2 : 3-benztriazole** (6-Nitro-1 : 2 : 3-benztriazole).

Long, pale yellow needles. M.p.  $211^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol. cold  $H_2O$ . Sublimes with partial decomp. Reacts acid. Undecomp. by acids or alkalis.

**N-Me**:  $C_7H_6O_2N_4$ . MW, 178. Cryst. from EtOH. M.p.  $163^\circ$ . Sol. hot  $C_6H_6$ ,  $H_2O$ , EtOH,  $CHCl_3$ , AcOH. Spar. sol. cold  $H_2O$ .

Nietzki, Hagenbach, *Ber.*, 1897, **30**, 544.

Zincke, *Ann.*, 1900, **311**, 290.

Pinnow, Koch, *Ber.*, 1897, **30**, 2852.

Macciotta, *Chem. Abstracts*, 1933, **27**, 4528.

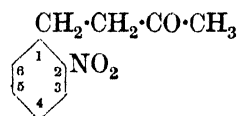
**N-Nitrobenzylacetamide**.

See under Nitrobenzylamine.

**N-Nitrobenzylacetanilide**.

See under Nitrobenzylaniline.

**o-Nitrobenzylacetone** (4-o-Nitrophenyl-butanone-2)



$C_{10}H_{11}O_3N$

MW, 193

Liq. B.p.  $183-5^\circ/13$  mm.  $Zn + NH_4OH \rightarrow$  tetrahydroquinaldine.

**Oxime**: cryst. M.p.  $97^\circ$ . Sol. EtOH, HCl.Aq.

**Semicarbazone**: powder. M.p.  $169-70^\circ$ . Insol.  $H_2O$ , EtOH.

Alber, *J. prakt. Chem.*, 1905, **71**, 45.

**p-Nitrobenzylacetone** (4-p-Nitrophenyl-butanone-2).

Needles from EtOH or  $Et_2O$ . M.p.  $42^\circ$  ( $40-1^\circ$ ). B.p.  $204^\circ/13$  mm. Sol. EtOH,  $Et_2O$ .

**Oxime**: needles or plates from EtOH. M.p.  $120^\circ$ . Sol. hot EtOH.

**Semicarbazone**: powder. M.p.  $198.5^\circ$ . Sol. acids. Prac. insol. EtOH. Insol.  $H_2O$ .

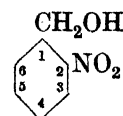
**Phenylhydrazone**: yellow cryst. M.p.  $103^\circ$ . Decomp. on keeping.

Alber, *J. prakt. Chem.*, 1905, **71**, 44.

Fichter, Wortsmann, *Ber.*, 1904, **37**, 1994.

Mech, *Compt. rend.*, 1906, **143**, 752.

**o-Nitrobenzyl Alcohol**



$C_7H_7O_3N$

MW, 153

Needles from  $H_2O$ . M.p.  $74^\circ$ . B.p.  $270^\circ$ ,  $168^\circ/20$  mm. Sol. EtOH,  $Et_2O$ .

**Benzoyl**: m.p.  $101-2^\circ$ .

Pierron, *Bull. soc. chim.*, 1901, **25**, 853.

Bamberger, *Ber.*, 1918, **51**, 609.

Schenck, *Ber.*, 1934, **67**, 1571.

Kalle, D.R.P., 104,360, (*Chem. Zentr.*, 1899, II, 950).



**m-Nitrobenzyl Alcohol.**

Needles from  $H_2O$ . M.p.  $27^\circ$ . B.p.  $175-80^\circ/3$  mm.  $D_{15}^{20} 1.296$ . Sol.  $H_2O$ , EtOH,  $Et_2O$ .

*Benzoyl*: m.p.  $71-2^\circ$ .

Grimaux, *Compt. rend.*, 1867, **65**, 211.

Becker, *Ber.*, 1882, **15**, 2090.

Thorp, Wildman, *J. Am. Chem. Soc.*, 1915, **37**, 373.

**p-Nitrobenzyl Alcohol.**

Cryst. from  $H_2O$ . M.p.  $93^\circ$ . B.p.  $185^\circ/12$  mm. Sol.  $H_2O$ , EtOH.

*Formyl*: cryst. from EtOH. M.p.  $31^\circ$ .

*Acetyl*: m.p.  $78^\circ$ .

*Propionyl*: cryst. from EtOH. M.p.  $33^\circ$ .

*Butyryl*: cryst. from EtOH. M.p.  $35^\circ$ .

*Palmityl*: cryst. from EtOH. M.p.  $42-42.5^\circ$ .

*Benzoyl*: m.p.  $94-5^\circ$ .

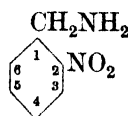
Dieffenbach, D.R.P., 214,949, (*Chem. Zentr.*, 1909, II, 1781).

Basler, *Ber.*, 1883, **16**, 2715.

Norris, Watt, Thomas, *J. Am. Chem. Soc.*, 1916, **38**, 1077.

Reid, *J. Am. Chem. Soc.*, 1917, **39**, 306.

**o-Nitrobenzylamine**



$C_7H_8O_2N_2$  MW, 152

Non-volatile liq. Sol.  $H_2O$ . Insol. conc. KOH. Absorbs  $CO_2$  from the air.

*N-Formyl*: *N-o*-nitrobenzylformamide. Cryst. from  $H_2O$ . M.p.  $88-90^\circ$ .

*N-Acetyl*: *N-o*-nitrobenzylacetamide. Needles. M.p.  $97-9^\circ$ .

*N-Benzoyl*: *N-o*-nitrobenzylbenzamide. Needles from EtOH. M.p.  $110^\circ$ .

*N-o-Toluy*: needles from EtOH. M.p.  $134-5^\circ$ .

*N-p-Toluy*: cryst. from EtOH. M.p.  $140-2^\circ$ .

*N-Benzenesulphonyl*: *N-o*-nitrobenzylbenzenesulphonamide. Yellow cryst. from  $C_6H_6$ . M.p.  $92^\circ$ .

*N-Me*:  $C_8H_{10}O_2N_2$ . MW, 166. Oil. Sol.  $H_2O$ . *B,HCl*: plates. M.p.  $175-176.5^\circ$ . *N-Acetyl*: m.p.  $57-8^\circ$ .

*N-Et*:  $C_9H_{12}O_2N_2$ . MW, 180. Oil. *B,HCl*: cryst. from EtOH. M.p.  $184-6^\circ$ .

*N-Di-Et*:  $C_{11}H_{16}O_2N_2$ . MW, 208. Yellow liq. B.p.  $175-7^\circ/42$  mm. Spar. sol.  $H_2O$ . *Picrate*: m.p.  $117^\circ$ .

*N-Allyl*:  $C_{10}H_{12}O_2N_2$ . MW, 192. Oil. Misc. with EtOH. Spar. sol.  $H_2O$ . *B,HCl*: needles from EtOH- $Et_2O$ . M.p.  $136-7^\circ$ .  $B_2H_2PtCl_6$ : yellow needles from EtOH- $Et_2O$ . M.p.  $163^\circ$ .

*N-Phenyl*: see *o*-Nitrobenzylaniline.

*Picrate*: yellow needles. M.p.  $206-8^\circ$ .

Wolff, *Ber.*, 1892, **25**, 3031.

Gabriel, Jansen, *Ber.*, 1891, **24**, 3092.

Noelting, Kregczy, *Bull. soc. chim.*, 1916, **19**, 335.

Ing, Manske, *J. Chem. Soc.*, 1926, 2348.

**m-Nitrobenzylamine.**

*N-Acetyl*: *N-m*-nitrobenzylacetamide. Needles. M.p.  $91^\circ$ .

*N-Benzenesulphonyl*: colourless needles or yellow prisms from  $C_6H_6$ . M.p.  $123-4^\circ$ .

*N-Me*: oil. *B,HCl*: needles from  $H_2O$ . M.p.  $181^\circ$ .

*N-Di-Et*: yellow liq. B.p.  $206-8^\circ/42$  mm. Sol. most org. solvents. Spar. sol.  $H_2O$ . *Picrate*: m.p.  $161^\circ$ .

*N-Phenyl*: see *m*-Nitrobenzylaniline.

*Benzylidene*: prisms. M.p.  $42^\circ$ .

*m-Nitrobenzylidene*: leaflets. M.p.  $141^\circ$ .

*p-Nitrobenzylidene*: needles. M.p.  $115^\circ$ .

See last two references above and also

Gabriel, Hendess, *Ber.*, 1887, **20**, 2869.

**p-Nitrobenzylamine.**

Needles. M.p.  $40^\circ$ . Strong base. Absorbs  $CO_2$  from the air.

*B,HCl*: m.p.  $222^\circ$ .

*N-Acetyl*: *N-p*-nitrobenzylacetamide. Needles from  $H_2O$ . M.p.  $133^\circ$ .

*N-Benzoyl*: *N-p*-nitrobenzylbenzamide. Needles from EtOH. M.p.  $155-6^\circ$ .

*N-Benzenesulphonyl*: *N-p*-nitrobenzylbenzenesulphonamide. Orange-yellow leaflets from  $C_6H_6$ . M.p.  $118^\circ$ .

*N-Me*: yellow oil. Sol. most org. solvents. *B,HCl*: needles from EtOH. M.p.  $226^\circ$ .

$B_2H_2PtCl_6$ : yellowish-red plates from EtOH.Aq. M.p.  $220^\circ$ .  $B_2(COOH)_2$ : needles from EtOH.Aq. M.p.  $188^\circ$ .

*N-Di-Me*:  $C_9H_{12}O_2N_2$ . MW, 180. Yellow oil. Volatile in steam.

*N-Et*: yellow oil. *B,HCl*: cryst. from EtOH.Aq. M.p.  $226^\circ$ .  $B_2(COOH)_2$ : plates from EtOH.Aq. M.p.  $207^\circ$ .

*N-Di-Et*: yellow liq. B.p.  $219-21^\circ/42$  mm. Sol. most org. solvents. Spar. sol.  $H_2O$ . *Picrate*: m.p.  $131^\circ$ .

*N-Propyl*:  $C_{10}H_{14}O_2N_2$ . MW, 194. *B,HCl*: needles from EtOH.Aq. M.p.  $225^\circ$ .  $B_2H_2PtCl_6$ : yellow needles from EtOH.Aq. M.p.  $177^\circ$ .

$B_2(COOH)_2$ : leaflets from EtOH.Aq. M.p.  $228^\circ$ .

*N-Isoamyl*:  $C_{12}H_{18}O_2N_2$ . MW, 222. *B,HCl*: prisms from EtOH- $Et_2O$ . M.p.  $204^\circ$ .  $B_2H_2PtCl_6$ : orange plates from EtOH.Aq.

M.p. 206°. *Picrate*: m.p. 144°.  $B_2(COOH)_2$ : leaflets from EtOH.Aq. M.p. 223°.

N-*Allyl*: yellow oil.  $B_2HCl$ : needles from EtOH-Et<sub>2</sub>O. M.p. 226°.  $B_2H_2PtCl_6$ : reddish-yellow prisms from EtOH. M.p. 174°. *Picrate*: needles. M.p. 146°.  $B_2(COOH)_2$ : leaflets from EtOH.Aq. M.p. 224°.

N-*Phenyl*: see p-Nitrobenzylaniline.

*Benzylidene*: prisms. M.p. 71°.

m-Nitrobenzylidene: plates. M.p. 115°.

p-Nitrobenzylidene: needles. M.p. 150°.

v. Braun, Deutsch, *Ber.*, 1912, **45**, 2197.

Curtius, *J. prakt. Chem.*, 1914, **89**, 526.

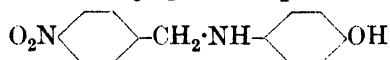
Noelting, Kregczy, *Bull. soc. chim.*, 1916, **19**, 335.

Salkowski, *Ber.*, 1889, **22**, 2142.

Paal, Sprenger, *Ber.*, 1897, **30**, 62.

Ing, Manske, *J. Chem. Soc.*, 1926, 2348.

#### N-4-Nitrobenzyl-p-aminophenol



$C_{13}H_{12}O_3N_2$

MW, 244

Reddish-brown cryst. from  $CHCl_3$  or  $C_6H_6$ , m.p. 114–15°, cryst. +  $1H_2O$  from solvents containing  $H_2O$ , m.p. 87–8°. Sol.  $Na_2CO_3$ .Aq.

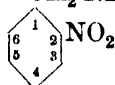
$B_2HCl$ : m.p. 191°.

O-*Benzoyl*: cryst. from  $C_6H_6$ . M.p. 218–20°.  $B_2HCl$ : m.p. 110–12°.

N-*Benzoyl*: yellowish needles. M.p. 208–10°.

Bakunin, Profilo, *Gazz. chim. ital.*, 1907, **37**, ii, 241.

#### o-Nitrobenzylaniline



$C_{13}H_{12}O_2N_2$

MW, 228

Needles from EtOH. Two forms: (i) m.p. 57°; (ii) m.p. 44° (metastable).  $Zn + HCl \rightarrow$  2-phenylindazole.  $Zn + AcOH \rightarrow$  o-amino-benzylaniline.

N-*Me*:  $C_{14}H_{14}O_2N_2$ . MW, 242. Red cryst. from EtOH. M.p. 72°. Spar. sol. ligroin.

N-*Et*:  $C_{15}H_{16}O_2N_2$ . MW, 256. Brown cryst. from EtOH. M.p. 66°. Sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH, Et<sub>2</sub>O. Insol. ligroin. *Hydrochloride*: m.p. about 158°. *Chloroplatinate*: orange ppt. M.p. 116–17° decomp.

N-*Formyl*: N-o-nitrobenzylformanilide. Yellow needles or plates from  $CS_2$ .

N-*Acetyl*: N-o-nitrobenzylacetanilide. Cryst. M.p. 75°. Spar. sol. ligroin.

N-*Benzoyl*: N-o-nitrobenzylbenzanilide. Cryst. M.p. 101°. Sol.  $CHCl_3$ . Spar. sol. EtOH, Et<sub>2</sub>O.

N-*Benzenesulphonyl*: needles from EtOH. M.p. 143°. Spar. sol. Et<sub>2</sub>O,  $C_6H_6$ .

N-*Nitroso*: prisms from Et<sub>2</sub>O. M.p. 84° (80.5–81°). Sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. ligroin.

Lellman, Stickel, *Ber.*, 1886, **19**, 1605.

Gabriel, *Ber.*, 1887, **20**, 2229.

#### m-Nitrobenzylaniline.

Orange-red needles from EtOH. M.p. 84°. Sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. Et<sub>2</sub>O.

$B_2HCl$ : m.p. 100–20°.

N-*Me*: yellow leaflets from EtOH. M.p. 51–2°. *Picrate*: m.p. 112–13°.

N-*Et*: reddish-brown prisms from EtOH. M.p. 69°. Very sol.  $CHCl_3$ ,  $C_6H_6$ . Sol. EtOH, Et<sub>2</sub>O. Insol. ligroin.  $B_2HCl$ : prisms from  $CHCl_3$ . M.p. 186°. *Picrate*: cryst. from  $CHCl_3$ -ligroin. M.p. 131°.

N-*Acetyl*: N-m-nitrobenzylacetanilide. Yellow needles from Et<sub>2</sub>O. M.p. 48°. Sol. EtOH, Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ .

Purgotti, Monti, *Gazz. chim. ital.*, 1900, **30**, ii, 256.

Reilly, Moore, Drum, *J. Chem. Soc.*, 1928, 563.

Gabriel, Hendess, *Ber.*, 1887, **20**, 2869.

#### p-Nitrobenzylaniline.

Orange prisms from EtOH.Aq. M.p. 72° (68°). Sol. Et<sub>2</sub>O,  $C_6H_6$ , hot EtOH. Fe + AcOH  $\rightarrow$  p-aminobenzylaniline.

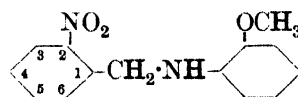
N-*Et*: yellow cryst. M.p. 67°. Very sol.  $CHCl_3$ ,  $C_6H_6$ . Sol. EtOH, Et<sub>2</sub>O. Insol. ligroin, pet. ether.

N-*Nitroso*: straw-yellow leaflets or needles. M.p. 75.5–76°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ , hot EtOH. Spar. sol. ligroin.

Höchst, D.R.P., 97,847, (*Chem. Zentr.*, 1898, II, 696).

Paal, Sprenger, *Ber.*, 1897, **30**, 69.

#### 2-Nitrobenzyl-o-anisidine



$C_{14}H_{14}O_3N_2$

MW, 258

Orange cryst. from EtOH. M.p. 80°. Sol. most org. solvents.

$B_2HCl$ : prisms from EtOH. M.p. 158°.

Paal, Schilling, *J. prakt. Chem.*, 1896, **54**, 277.

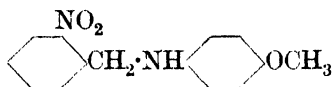
**4-Nitrobenzyl-*o*-anisidine.**

Red cryst. from EtOH. M.p. 95°. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

*N*-Formyl: yellowish plates from EtOH. M.p. 102°.

*N*-Acetyl: yellowish plates from EtOH. M.p. 78°.

Paal, Benker, *Ber.*, 1899, 32, 1253.

**2-Nitrobenzyl-*p*-anisidine**

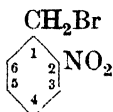
C<sub>14</sub>H<sub>14</sub>O<sub>3</sub>N<sub>2</sub> MW, 258

Red leaflets from EtOH.Aq. M.p. 73°. Sol. most org. solvents. Spar. sol. ligroin.

*B.HCl*: needles from EtOH-Et<sub>2</sub>O. M.p. 185°.

*N*-Formyl: cryst. from EtOH. M.p. 69°.

Paal, Schilling, *J. prakt. Chem.*, 1896, 54, 283.

***o*-Nitrobenzyl bromide**

C<sub>7</sub>H<sub>6</sub>O<sub>2</sub>NBr MW, 216

Cryst. from EtOH.Aq. M.p. 46-7°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Darkens on exposure to light.

Norris, Watt, Thomas, *J. Am. Chem. Soc.*, 1916, 38, 1077.

Opolski, Kowalski, Pilewski, *Ber.*, 1916, 49, 2282.

***m*-Nitrobenzyl bromide.**

Needles or plates. M.p. 58-9°.

See first reference above and also

Wachendorff, *Ann.*, 1877, 185, 277.

***p*-Nitrobenzyl bromide.**

Needles from EtOH. M.p. 99-100°. Easily sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. cold H<sub>2</sub>O.

Brewster, *J. Am. Chem. Soc.*, 1918, 40, 406.

Lyons, Reid, *J. Am. Chem. Soc.*, 1917, 39, 1727.

Mourev, Brown, *Bull. soc. chim.*, 1921, 29, 1006.

See also previous references.

***o*-Nitrobenzyl chloride**

C<sub>7</sub>H<sub>6</sub>O<sub>2</sub>NCl

MW, 171.5

Cryst. from ligroin. M.p. 48-9°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Kumpf, *Ann.*, 1884, 224, 100.

Noelting, *Ber.*, 1884, 17, 385.

Holleman, *Rec. trav. chim.*, 1914, 33, 12.

***m*-Nitrobenzyl chloride.**

Pale yellow needles from ligroin. M.p. 45-7° (44-8°). B.p. 173-83°/30-5 mm. Volatile in steam. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Gabriel, Borgmann, *Ber.*, 1883, 16, 2064.

Abelli, *Gazz. chim. ital.*, 1883, 13, 98.

Holleman, *Rec. trav. chim.*, 1914, 33, 12.

Norris, Taylor, *J. Am. Chem. Soc.*, 1924, 46, 753.

***p*-Nitrobenzyl chloride.**

Plates or needles from EtOH M.p. 71°. Sol. EtOH, Et<sub>2</sub>O.

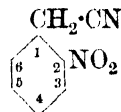
Beilstein, Gietner, *Ann.*, 1886, 139, 337.

Noelting, *Ber.*, 1884, 17, 385.

Alway, *J. Am. Chem. Soc.*, 1902, 24, 1062.

Norris, Taylor, *J. Am. Chem. Soc.*, 1924, 46, 753.

***o*-Nitrobenzyl cyanide** (*o*-Nitrophenylacetonitrile)



C<sub>8</sub>H<sub>6</sub>O<sub>2</sub>N<sub>2</sub> MW, 162

Prisms from AcOH or EtOH, needles from H<sub>2</sub>O. M.p. 84°. B.p. 178°/12 mm. Sol. H<sub>2</sub>O. Spar. sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Alkalis → deep bluish-violet col.

Salkowski, *Ber.*, 1884, 17, 507.

Bamberger, *Ber.*, 1886, 19, 2635.

Reissert, *Ber.*, 1908, 41, 3814.

Pschorr, Hoppe, *Ber.*, 1910, 43, 2547.

***m*-Nitrobenzyl cyanide** (*m*-Nitrophenylacetonitrile).

Cryst. from Et<sub>2</sub>O-ligroin. M.p. 61-2°. Sol. CHCl<sub>3</sub>. Mod. sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin.

Haller, *Ann.*, 1908, 358, 357.

Salkowski, *Ber.*, 1884, 17, 506.

***p*-Nitrobenzyl cyanide** (*p*-Nitrophenylacetonitrile).

Plates. M.p. 116-17°. Mod. sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Sol. alc. KOH with intense carmine col.

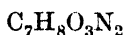
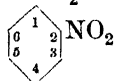
Robertson, *Organic Syntheses*, Collective Vol. I, 389.

Bucklow, *Ber.*, 1900, 33, 170.

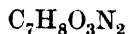
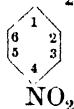
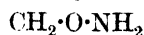
Gabriel, *Ber.*, 1881, 14, 2342.

**N-Nitrobenzylformanilide.**

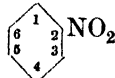
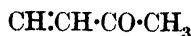
See under Nitrobenzylaniline.

**N-2-Nitrobenzylhydroxylamine**

MW, 168

Needles from  $\text{C}_6\text{H}_6$ . M.p.  $70^\circ$ . At  $140^\circ$  becomes dark blue in col.*B, HCl*: needles. M.p.  $185^\circ$  decomp.Kjellin, Kuylenstjerna, *Ber.*, 1897, 30, 517.**N-3-Nitrobenzylhydroxylamine.**Needles from  $\text{H}_2\text{O}$ . M.p.  $79.5\text{--}80.5^\circ$ .*B, HCl*: m.p.  $145\text{--}6^\circ$ .Behrend, *Ann.*, 1891, 265, 245.**N-4-Nitrobenzylhydroxylamine.**Needles from  $\text{H}_2\text{O}$ . M.p.  $120\text{--}5^\circ$ . Sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Insol. ligroin. Reduces Fehling's.*B, HCl*: needles. M.p.  $180\text{--}2^\circ$  decomp.Behrend, Leuchs, *Ann.*, 1890, 257, 243.Behrend, König, *Ann.*, 1891, 263, 193.**O-4-Nitrobenzylhydroxylamine**

MW, 168

Prisms from  $\text{C}_6\text{H}_6$ -ligroin. M.p.  $56^\circ$ . Does not reduce Fehling's.*B, HCl*: leaflets from EtOH. M.p.  $217^\circ$ . Part. hydrolysed by boiling  $\text{H}_2\text{O}$ .Brady, Klein, *J. Chem. Soc.*, 1927, 881.**o-Nitrobenzylideneacetone** (*o*-Nitrobenzalacetone, methyl *o*-nitrostyryl ketone)

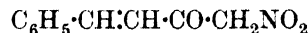
MW, 191

Needles from  $\text{Et}_2\text{O}$  or EtOH.Aq. M.p.  $60^\circ$  ( $58\text{--}9^\circ$ ). Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol. ligroin.  $\text{SnCl}_2 + \text{HCl} \rightarrow$  quinaldine. Dil.  $\text{HOCl} \rightarrow$  *o*-nitrocinnamic acid.Drewsen, *Ber.*, 1883, 16, 1954.Fischer, Kuzel, *ibid.*, 36.

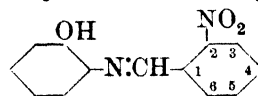
M.L.B., D.R.P., 20,255.

**m-Nitrobenzylideneacetone** (*m*-Nitrobenzalacetone, methyl *m*-nitrostyryl ketone).Prisms from AcOH. M.p.  $94\text{--}5^\circ$ .*Phenylhydrazone*: m.p.  $155^\circ$ .Vorländer, *Ann.*, 1897, 294, 293.Ruhemann, *J. Chem. Soc.*, 1903, 83, 1375.**p-Nitrobenzylideneacetone** (*p*-Nitrobenzalacetone, methyl *p*-nitrostyryl ketone).M.p.  $110^\circ$ . Sol. conc.  $\text{H}_2\text{SO}_4$  with yellowish-red col.*Phenylhydrazone*: red cryst. from EtOH. M.p.  $195\text{--}6^\circ$ .*p*-Chlorophenylhydrazone: m.p.  $218\text{--}19^\circ$ .*o*-Tolylhydrazone: m.p.  $180\text{--}1^\circ$ .

M.L.B., D.R.P., 20,255.

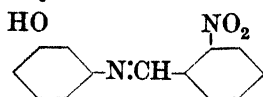
Baeyer, Becker, *Ber.*, 1883, 16, 1969.Auwers, Voss, *Ber.*, 1909, 42, 4425. **$\omega$ -Nitrobenzylideneacetone** ( $\omega$ -Nitrobenzalacetone)

MW, 191

Yellowish plates from EtOH. M.p.  $97\text{--}8^\circ$  after sintering at  $83^\circ$ . Sol. hot alkalis with yellow col.Harries, *Ann.*, 1901, 319, 254.**2-Nitrobenzylidene-o-aminophenol**

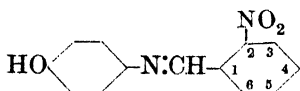
MW, 242

Yellow needles from  $\text{C}_6\text{H}_6$ . M.p.  $107^\circ$  ( $104^\circ$ ). Sol. most org. solvents. Slowly darkens on exposure to light.*Me ether*: see 2-Nitrobenzylidene-o-anisidine.Levi, *Gazz. chim. ital.*, 1929, 59, 544.Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1920.**3-Nitrobenzylidene-o-aminophenol.**Yellowish needles from EtOH. M.p.  $135^\circ$ . Sol. most org. solvents.Pope, *J. Chem. Soc.*, 1908, 93, 535.**4-Nitrobenzylidene-o-aminophenol.**Yellow needles from  $\text{C}_6\text{H}_6$  or  $\text{CCl}_4$ . M.p.  $158\text{--}60^\circ$  ( $154^\circ$ ). Sol. EtOH, AcOEt, toluene. Insol. pet. ether, ligroin. Alc. NaOH  $\rightarrow$  red col.*Me ether*: see 4-Nitrobenzylidene-o-anisidine.Levi, *Gazz. chim. ital.*, 1929, 59, 544.Pope, *J. Chem. Soc.*, 1908, 93, 536.Möhlau, Adams, *Chem. Zentr.*, 1907, I, 108.

2-Nitrobenzylidene-*m*-aminophenol $C_{13}H_{10}O_3N_2$ 

MW, 242

Yellow prisms. M.p. 106–7°.

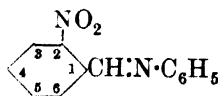
*Me ether*: see 2-Nitrobenzylidene-*m*-anisidine.Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1921.2-Nitrobenzylidene-*p*-aminophenol $C_{13}H_{10}O_3N_2$ 

MW, 242

Yellowish needles from EtOH.Aq. M.p. 163° (159°).

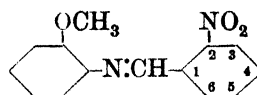
*Me ether*: see 2-Nitrobenzylidene-*p*-anisidine.*B, HCl*: m.p. 216°.Pope, Fleming, *J. Chem. Soc.*, 1908, 93, 1918.Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1921.3-Nitrobenzylidene-*p*-aminophenol.

Brown needles from EtOH.Aq. M.p. 154°.

Pope, *J. Chem. Soc.*, 1908, 93, 534.4-Nitrobenzylidene-*p*-aminophenol.Yellow prisms from Et<sub>2</sub>O. M.p. 168°. Sol. most org. solvents. Insol. ligroin.*Me ether*: see 4-Nitrobenzylidene-*p*-anisidine.*Et ether*: see 4-Nitrobenzylidene-*p*-phenetidine.Pope, *J. Chem. Soc.*, 1908, 93, 533.*o*-Nitrobenzylideneaniline (*o*-Nitrobenzaldehyde anil) $C_{13}H_{10}O_2N_2$ 

MW, 226

Yellow leaflets from 80% EtOH. M.p. 69.5°. B.p. 220°/15 mm.

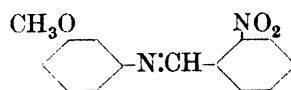
Knoevenagel, *Ber.*, 1898, 31, 2609 (*Footnote*).*m*-Nitrobenzylideneaniline (*m*-Nitrobenzaldehyde anil).Needles from EtOH. M.p. 66° (61°). Sol. AcOH.  $NH_2OH \rightarrow m$ -nitrobenz-anti-aldoxime.Lazorenko, *Jahresber. Fortschr. Chem.*, 1870, 760.Schwalbe, *Chem. Zentr.*, 1903, I, 231.*p*-Nitrobenzylideneaniline (*p*-Nitrobenzaldehyde anil).Yellowish leaflets from Et<sub>2</sub>O. M.p. 93°.Fischer, *Ber.*, 1881, 14, 2525.2-Nitrobenzylidene-*o*-anisidine $C_{14}H_{12}O_3N_2$ 

MW, 256

Yellow cryst. M.p. 64–5°.

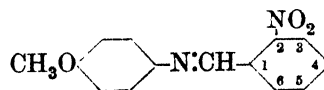
Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1921.4-Nitrobenzylidene-*o*-anisidine.

Yellow plates from EtOH.Aq. M.p. 111°.

*B, HCl*: m.p. 141°.Pope, Fleming, *J. Chem. Soc.*, 1908, 93, 1917.2-Nitrobenzylidene-*m*-anisidine $C_{14}H_{12}O_3N_2$ 

MW, 256

Yellow cryst. M.p. 74–5°.

Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1921.2-Nitrobenzylidene-*p*-anisidine $C_{14}H_{12}O_3N_2$ 

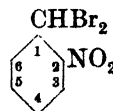
MW, 256

Yellow cryst. M.p. 80–1°.

See previous reference.

4-Nitrobenzylidene-*p*-anisidine

Yellow leaflets from EtOH. M.p. 139°.

*B, HCl*: m.p. 200°.Pope, Fleming, *J. Chem. Soc.*, 1908, 93, 1917.*o*-Nitrobenzylidene bromide $C_7H_5O_2NBr_2$ 

MW, 295

Prisms from EtOH. M.p. 46°. Boiling alkalis  $\rightarrow o$ -nitrobenzaldehyde.Reissert, *Ber.*, 1897, 30, 1043.

**m-Nitrobenzylidene bromide.**

Needles from EtOH. M.p. 101–2°.

Wachendorff, *Ann.*, 1877, **185**, 278.

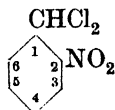
**p-Nitrobenzylidene bromide.**

Needles from EtOH. M.p. 82°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Ox. → p-nitrobenzoic acid. Heat with aniline → pararosaniline.

Reinhardt, *Ber.*, 1913, **46**, 3598.

See also previous reference.

**o-Nitrobenzylidene chloride**



C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>NCl<sub>2</sub>

MW, 206

Cryst. from EtOH. M.p. 27°. B.p. 143–4°/12 mm. Turns brown when exposed to light. Decomp. on long standing in air. Warm conc. HCl → o-nitrobenzaldehyde.

Kliegl, *Ber.*, 1907, **40**, 4939.

Kliegl, Haas, *Ber.*, 1911, **44**, 1214.

**m-Nitrobenzylidene chloride.**

Cryst. from EtOH. M.p. 65°. Sol. hot EtOH, Et<sub>2</sub>O. Red. → m-toluidine.

Widman, *Ber.*, 1880, **13**, 676.

Kliegl, Haas, *Ber.*, 1909, **42**, 2585.

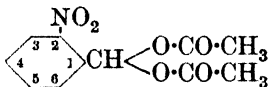
Holleman, *Rec. trav. chim.*, 1914, **33**, 18.

**p-Nitrobenzylidene chloride.**

Prisms from EtOH. M.p. 46° (42°). Sol. EtOH, Et<sub>2</sub>O.

See last two references above.

**o-Nitrobenzylidene diacetate**



C<sub>11</sub>H<sub>11</sub>O<sub>6</sub>N

MW, 253

Prisms from ligroin. M.p. 90° (87–8°). Sol. most org. solvents.

Thiele, Winter, *Ann.*, 1900, **311**, 356.

Baeyer, D.R.P., 121,788, (*Chem. Zentr.*, 1901, II, 70).

Bakunin, Parlati, *Gazz. chim. ital.*, 1906, **36**, 265.

Bakunin, Fisceman, *Gazz. chim. ital.*, 1916, **46**, 93.

**m-Nitrobenzylidene diacetate.**

Cryst. from EtOH. M.p. 72°. Sol. most org. solvents.

See last reference above and also

Bakunin, Parlati, *Gazz. chim. ital.*, 1906, **36**, 266.

**p-Nitrobenzylidene diacetate.**

Prisms from EtOH. M.p. 127° (125°). Sol. most org. solvents.

Thiele, Winter, *Ann.*, 1900, **311**, 355.

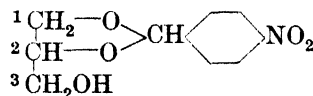
Baeyer, D.R.P., 121,788, (*Chem. Zentr.*, 1901, II, 70).

Bakunin, Parlati, *Gazz. chim. ital.*, 1906, **36**, 266.

• Kohler, Reimer, *Am. Chem. J.*, 1904, **31**, 169.

Bakunin, Fisceman, *Gazz. chim. ital.*, 1916, **46**, 93.

**1 : 2-p-Nitrobenzylideneglycerol**



C<sub>10</sub>H<sub>11</sub>O<sub>5</sub>N

MW, 225

Liq. B.p. 177–9°/0.3 mm. HCl (gas) at 100° → equilibrium mixture with the 1 : 3-isomer in ratio 2 : 1. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, ligroin. Two series of derivatives known which are geometrical isomers.

*Me ether* : C<sub>11</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 239. *α-Form* : yellow prisms from MeOH. M.p. 47°. Sol. most org. solvents. Insol. H<sub>2</sub>O, ligroin. *β-Form* : feathery cryst. M.p. 42°. Somewhat more sol. than *α*-isomer.

*Benzoyl* : *α-form*, greenish-yellow cryst. from AcOEt. M.p. 115°. *β-Form* : cryst. from EtOH. M.p. 178°.

*p-Nitrobenzoyl* : *α-form*, straw-yellow needles from toluene. M.p. 117–18°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. *β-Form* : cryst. from toluene. M.p. 110°.

Hibbert, Sturrock, *J. Am. Chem. Soc.*, 1928, **50**, 3374.

Hibbert, Carter, *ibid.*, 3376.

Hibbert, Platt, Carter, *J. Am. Chem. Soc.*, 1929, **51**, 3641.

**1 : 3-p-Nitrobenzylideneglycerol.**

Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 88°. On standing m.p. rises slowly to 98° although recryst. from C<sub>6</sub>H<sub>6</sub> invariably gives the comp. m.p. 88°. HCl (gas) at 100° → equilibrium mixture with the 1 : 2-isomer in ratio 1 : 2. Forms two series of derivs. which are geometrical isomers.

*Me ether* : *α-form*, grayish needles from EtOH. M.p. 139°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, ligroin. Insol. H<sub>2</sub>O. *β-Form* : needles from EtOH. M.p. 106°. Slightly more sol. than the *α-form*.

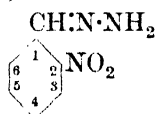
*Benzoyl* : *α-form*, plates from AcOEt. M.p. 204° Sol. Et<sub>2</sub>O, AcOEt, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, hot

ligroin. Insol.  $H_2O$ .  $\beta$ -Form: m.p.  $159^\circ$ . Rather more sol. than  $\alpha$ -form.

*p*-Nitrobenzoyl:  $\alpha$ -form, spicules from toluene. M.p.  $208^\circ$ .  $\beta$ -Form: needles from toluene. M.p.  $202^\circ$ .

See previous references.

**o-Nitrobenzylidenhydrazine** (*o*-Nitrobenzaldehyde hydrazone)



$C_7H_7O_2N_3$  MW, 165

Yellow prisms from EtOH. M.p.  $76^\circ$ . Very sol. most org. solvents.

Curtius, Lublin, *Ber.*, 1900, **33**, 2463.

***m*-Nitrobenzylidenhydrazine** (*m*-Nitrobenzaldehyde hydrazone).

Yellow plates from EtOH. M.p.  $107^\circ$ . Mod. sol. org. solvents.

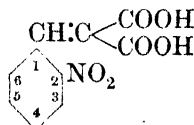
See previous reference.

***p*-Nitrobenzylidenhydrazine** (*p*-Nitrobenzaldehyde hydrazone).

Orange prisms from EtOH. M.p.  $134^\circ$ .

See previous reference.

**o-Nitrobenzylidenemalonic Acid**



$C_{10}H_7O_6N$  MW, 237

Needles from  $H_2O$ . M.p.  $162^\circ$ . Sol.  $H_2O$ ,  $Et_2O$ . Spar. sol.  $CHCl_3$ . Insol.  $CS_2$ ,  $C_6H_6$ .  $FeSO_4 + NH_4OH \rightarrow$  carbostyryl-3-carboxylic acid. Boiling with  $H_2O \rightarrow$  *o*-nitrobenzaldehyde,  $CO_2$ , malonic and *o*-nitrocinnamic acids.

*Di-Me ester*:  $C_{12}H_{11}O_6N$ . MW, 265. M.p.  $65-6^\circ$ .

*Di-Et ester*:  $C_{14}H_{15}O_6N$ . MW, 293. M.p.  $53^\circ$ .

*Mononitrile*: *o*-nitro- $\alpha$ -cyanocinnamic acid.  $C_{10}H_6O_4N_2$ . MW, 218. Yellow cryst. from AcOH. M.p.  $226-8^\circ$  ( $223^\circ$ ). Sol. EtOH. *Me ester*:  $C_{11}H_8O_4N_2$ . MW, 232. Needles from EtOH. M.p.  $142^\circ$ . *Et ester*:  $C_{12}H_{10}O_4N_2$ . MW, 246. Yellow plates from EtOH. M.p.  $96^\circ$ . Sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. cold EtOH, ligroin. Prac. insol. hot  $H_2O$ . *Amide*:  $C_{10}H_7O_3N_3$ . MW, 217. Needles from dil. EtOH. M.p.  $173-4^\circ$ . Sol.  $H_2O$ , EtOH,  $Me_2CO$ . Spar. sol.  $Et_2O$ .

*Di-nitrile*:  $C_{10}H_5O_2N_3$ . MW, 199. Needles

from EtOH or AcOH. M.p.  $137.5-138^\circ$ . Sol.  $Me_2CO$ ,  $CHCl_3$ . Spar. sol.  $Et_2O$ , ligroin.

Meyer, *Monatsh.*, 1907, **28**, 53.

Stuart, *J. Chem. Soc.*, 1885, **47**, 155.

Heller, Wunderlich, *Ber.*, 1914, **47**, 1621, 2890.

Sudborough, Lloyd, *J. Chem. Soc.*, 1898, **73**, 88.

Issoglio, *Chem. Zentr.*, 1904, **I**, 878.

Baker, Eccles, *J. Chem. Soc.*, 1927, 2127.

Fiquet, *Ann. chim. phys.*, 1893, **29**, 491.

***m*-Nitrobenzylidenemalonic Acid.**

Cryst. M.p.  $209-10^\circ$  ( $205^\circ$ ) decomp. Sol. hot  $H_2O$  (part. decomp.). Spar. sol.  $Et_2O$ , cold  $H_2O$ . Boiling with  $H_2O \rightarrow CO_2$ , *m*-nitrobenzaldehyde, malonic and *m*-nitrocinnamic acids.

*Di-Me ester*: m.p.  $99-100^\circ$ .

*Di-Et ester*: plates from EtOH. M.p.  $75-6^\circ$  ( $73^\circ$ ).

*Mononitrile*: *m*-nitro- $\alpha$ -cyanocinnamic acid. Needles from EtOH.Aq. or  $Me_2CO$ . M.p.  $173-5^\circ$  ( $172^\circ$ ). *Me ester*: needles. M.p.  $135-6^\circ$ . *Et ester*: (i) colourless form. Prisms from EtOH or needles from  $EtOH-C_6H_6$ . M.p.  $135^\circ$  ( $127-8^\circ$ ). Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. ligroin. Insol.  $H_2O$ . (ii) Yellow form. Yellow cryst. from EtOH or  $C_6H_6$ . M.p.  $135^\circ$ .

Kötze, Kempe, *J. prakt. Chem.*, 1907, **75**, 507.

Sudborough, Lloyd, *J. Chem. Soc.*, 1898, **73**, 88.

Bertini, *Gazz. chim. ital.*, 1901, **31**, 275.

Baker, Eccles, *J. Chem. Soc.*, 1927, 2127.

Boehm, *Arch. Pharm.*, 1929, **267**, 702.

***p*-Nitrobenzylidenemalonic Acid.**

Cryst. M.p.  $227^\circ$  decomp. Heat  $\rightarrow$  *p*-nitrocinnamic acid +  $CO_2$ . Boiling with  $H_2O \rightarrow$  *p*-nitrobenzaldehyde,  $CO_2$ , malonic and *p*-nitrocinnamic acids.

*Di-Me ester*: needles from EtOH. M.p.  $136-7^\circ$ .

*Di-Et ester*: needles from EtOH. M.p.  $93^\circ$ . Sol. hot EtOH, ligroin,  $C_6H_6$ . Spar. sol.  $Et_2O$ .

*Mononitrile*: *p*-nitro- $\alpha$ -cyanocinnamic acid. Plates from EtOH. M.p.  $208^\circ$ . *Et ester*: needles. M.p.  $169-70^\circ$ . Sol. EtOH.

Knoevenagel, *Ber.*, 1898, **31**, 2593, 2613.

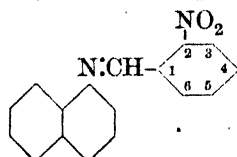
Stuart, *J. Chem. Soc.*, 1883, **43**, 408; 1885, **47**, 158.

Fiquet, *Ann. chim. phys.*, 1893, **29**, 489.

Boehm, *Arch. Pharm.*, 1929, **267**, 702.

Baker, Eccles, *J. Chem. Soc.*, 1927, 2127.

***o*-Nitrobenzylidene-1-naphthylamine**



$C_{17}H_{12}O_2N_2$

MW, 276

Yellow solid. M.p. 118–118.5°.

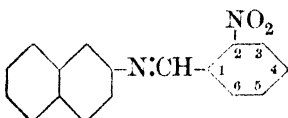
Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1923.

***m*-Nitrobenzylidene-1-naphthylamine.**

Yellow plates from ligroin. M.p. 102–3°. Sol.  $Et_2O$ ,  $CHCl_3$ . Spar. sol. ligroin.

Zenoni, *Gazz. chim. ital.*, 1893, 23, ii, 222, 519.

***o*-Nitrobenzylidene-2-naphthylamine**



$C_{17}H_{12}O_2N_2$

MW, 276

Yellow cryst. from pet. ether. M.p. 95–6° (91°).

Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1924.

***m*-Nitrobenzylidene-2-naphthylamine.**

Yellow plates from  $EtOH$ . M.p. 90°. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $EtOH$ .

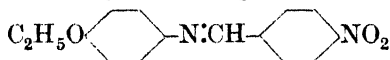
Haase, *Ber.*, 1903, 36, 593.

***p*-Nitrobenzylidene-2-naphthylamine.**

Yellow needles. M.p. 120–1°. Very sol.  $Et_2O$ ,  $Me_2CO$ . Mod. sol.  $EtOH$ , ligroin.

Zenoni, *Gazz. chim. ital.*, 1893, 23, ii, 223, 519.

**4-Nitrobenzylidene-*p*-phenetidine**



$C_{15}H_{14}O_3N_2$

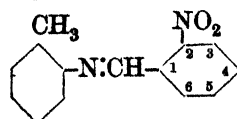
MW, 270

Yellow, needles from  $EtOH$ . M.p. 130.5°.

$B, HCl$ : m.p. 196°.

Pope, Fleming, *J. Chem. Soc.*, 1908, 93, 1917.

**2-Nitrobenzylidene-*o*-toluidine**



$C_{14}H_{12}O_2N_2$

MW, 240

Yellow cryst. M.p. 81–81.5°.

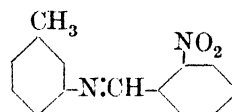
Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1918.

**3-Nitrobenzylidene-*o*-toluidine.**

Yellow prisms from  $EtOH$ . M.p. 78–9°. Very sol.  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ . Sol.  $EtOH$ .

Ruhemann; Watson, *J. Chem. Soc.*, 1904, 85, 1179.

**2-Nitrobenzylidene-*m*-toluidine**



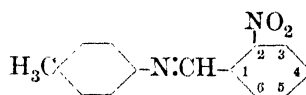
$C_{14}H_{12}O_2N_2$

MW, 240

Yellow solid. M.p. 52–3°.

Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1918.

**2-Nitrobenzylidene-*p*-toluidine**



$C_{14}H_{12}O_2N_2$

MW, 240

Yellow solid. M.p. 73–4°.

Senier, Clarke, *J. Chem. Soc.*, 1914, 105, 1919.

**3-Nitrobenzylidene-*p*-toluidine.**

Yellow needles. M.p. 96°. Sol.  $C_6H_6$ . Spar. sol.  $EtOH$ .

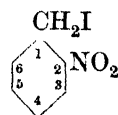
Ullmann, *Ber.*, 1903, 36, 1024.

**4-Nitrobenzylidene-*p*-toluidine.**

Yellow leaflets. M.p. 124.5°. Sol.  $Et_2O$ ,  $C_6H_6$ , hot  $EtOH$ .

Ullmann, *Ber.*, 1903, 36, 1022.

***o*-Nitrobenzyl iodide**



$C_7H_6O_2NI$

MW, 265

Plates. M.p. 75°.

Kumpf, *Ann.*, 1884, 224, 103.

***m*-Nitrobenzyl iodide.**

Yellow cryst. M.p. 84.5–86°.

Poggi, *Atti accad. Lincei*, 1925, 2, 423.

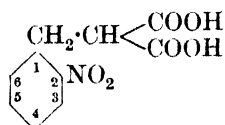


**p-Nitrobenzyl iodide.**

Needles from EtOH. M.p. 127° (124°). Spar. sol. cold EtOH.

Finkelstein, *Ber.*, 1910, **43**, 1531.  
Knoll, D.R.P., 230,172, (*Chem. Zentr.*, 1911, I, 359).  
Kumpf, *Ann.*, 1884, **224**, 99.

**o-Nitrobenzylmalonic Acid**



$C_{10}H_8O_6N$  MW, 239

Needles from  $H_2O$ . M.p. 164° decomp. Sol. MeOH, EtOH,  $Et_2O$ , AcOH, hot  $H_2O$ . Insol.  $CHCl_3$ ,  $C_6H_6$ , ligroin.

*Diamide*:  $C_{10}H_{11}O_4N_3$ . MW, 237. Needles from  $H_2O$ . M.p. 234° decomp. Sol. AcOEt, hot EtOH.

Reissert, *Ber.*, 1896, **29**, 634, 644.  
Baker, Eccles, *J. Chem. Soc.*, 1927, 2126.

**m-Nitrobenzylmalonic Acid.**

Cryst. from  $H_2O$ . M.p. 171° (164°) decomp.

*Diamide*: cryst. from  $H_2O$ . M.p. 203°.

*Amide-nitrile*:  $C_{10}H_9O_3N_3$ . MW, 219. Needles from EtOH. M.p. 147-8°. Sol.  $H_2O$ , EtOH,  $Me_2CO$ ,  $CHCl_3$ . Spar. sol.  $Et_2O$ .

Issoglio, *Chem. Zentr.*, 1904, I, 878.  
Gulland, Haworth, Virden, Callow, *J. Chem. Soc.*, 1929, 1666.  
Baker, Eccles, *J. Chem. Soc.*, 1927, 2127.

**p-Nitrobenzylmalonic Acid.**

Yellow powder. Decomp. at 100°.

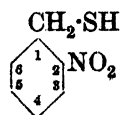
*Di-Me ester*:  $C_{12}H_{13}O_6N$ . MW, 267. Cryst. from MeOH. M.p. 82.5-83.5°.

*Di-Et ester*:  $C_{14}H_{17}O_6N$ . MW, 295. Prisms from EtOH.Aq. M.p. 168.5°. Sol. EtOH,  $Me_2CO$ . Mod. sol. warm  $H_2O$ . Spar. sol.  $Et_2O$ .

*Monohydrazide*: m.p. 137°.

Lellmann, Schleich, *Ber.*, 1887, **20**, 434.  
Reissert, *Ber.*, 1896, **29**, 635.  
Curtius, *J. prakt. Chem.*, 1930, **125**, 139.  
Baker, Eccles, *J. Chem. Soc.*, 1927, 2128.

**o-Nitrobenzyl Mercaptan**



$C_7H_7O_2NS$

MW, 169

Yellow needles from MeOH or AcOEt. M.p. 42-4° (47°, 29.5°). B.p. 149°/15 mm.

Gabriel, Stelzner, *Ber.*, 1896, **29**, 161.  
Price, Twiss, *J. Chem. Soc.*, 1909, **95**, 1727.

**m-Nitrobenzyl Mercaptan.**

Yellowish-white needles. M.p. 14° (11-12°). B.p. 164°/18 mm.

See last reference above and also  
Lutter, *Ber.*, 1897, **30**, 1068.

**p-Nitrobenzyl Mercaptan.**

Yellow needles or plates from EtOH. M.p. 58° (55°, 52.5°, 51°). B.p. 164°/15 mm.

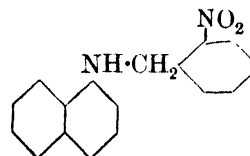
*Carbamate*: m.p. 140-1°.

Price, Twiss, *J. Chem. Soc.*, 1909, **95**, 1727.

Poggi, *Atti accad. Lincei*, 1925, **2**, 424

Horn, *J. Am. Chem. Soc.*, 1921, **43**, 2607.

**o-Nitrobenzyl-1-naphthylamine**



$C_{17}H_{14}O_2N_2$  MW, 278

Yellow needles from EtOH. M.p. 97°. Sol.  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ . Spar. sol. EtOH, ligroin. Insol.  $H_2O$ .

*N-Acetyl*: *N*-o-nitrobenzyl-1-acetnaphthalide. Leaflets from EtOH. M.p. 130°.

Darier, Mannassewitch, *Bull. soc. chim.*, 1902, **27**, 1057.

**m-Nitrobenzyl-1-naphthylamine.**

Orange-yellow prisms from EtOH. M.p. 94°. Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*N-Acetyl*: yellow needles from EtOH. M.p. 109-10°.

See previous reference.

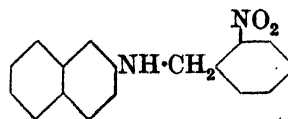
**p-Nitrobenzyl-1-naphthylamine.**

Orange leaflets from EtOH. M.p. 126-7°. Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. EtOH, ligroin.

*N-Acetyl*: *N*-p-nitrobenzyl-1-acetnaphthalide. Needles from EtOH. M.p. 112-13°.

See previous reference.

**o-Nitrobenzyl-2-naphthylamine**



$C_{17}H_{14}O_2N_2$

MW, 278

Red leaflets from EtOH-AcOH. M.p. 162°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Spar. sol. most org. solvents.

**N-Acetyl**: *N*-o-nitrobenzyl-2-acetnaphthalide. Prisms from EtOH. M.p. 117-18°.

**N-Nitroso**: yellow leaflets from EtOH. M.p. 102°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Very spar. sol. ligroin.

Darier, Mannassewitch, *Bull. soc. chim.*, 1902, 27, 1057.

Busch, Brand, *J. prakt. Chem.*, 1895, 52, 410.

**m-Nitrobenzyl-2-naphthylamine.**

Needles from EtOH. M.p. 80°.

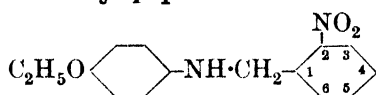
Darier, Mannassewitch, *Bull. soc. chim.*, 1902, 27, 1060.

**p-Nitrobenzyl-2-naphthylamine.**

Yellow leaflets from EtOH.Aq. M.p. 121.5°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH.

See previous reference.

**2-Nitrobenzyl-p-phenetidine**



C<sub>15</sub>H<sub>16</sub>O<sub>3</sub>N<sub>2</sub> MW, 272

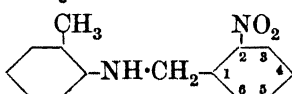
Red plates from EtOH.Aq. M.p. 52°. Sol. most org. solvents. Spar. sol. ligroin.

**B,HCl**: needles from EtOH-HCl. M.p. 163°.

**N-Formyl**: plates from C<sub>6</sub>H<sub>6</sub>. M.p. 96°.

Paal, Küttner, *J. prakt. Chem.*, 1893, 48, 555.

**2-Nitrobenzyl-o-toluidine**



C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub> MW, 242

Cryst. from EtOH. M.p. 96°. D<sub>15</sub> 1.278. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

**N-Formyl**: yellow needles from EtOH.Aq. M.p. 76°.

**N-Nitroso**: needles from EtOH. M.p. 64-5°.

Lellmann, Mayer, *Ber.*, 1892, 25, 3582.

**3-Nitrobenzyl-o-toluidine.**

Cryst. from EtOH. M.p. 62°. Sol. CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O, pet. ether.

Purgotti, Monti, *Gazz. chim. ital.*, 1900, 30, ii, 258.

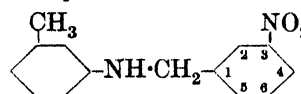
**4-Nitrobenzyl-o-toluidine.**

Red needles. M.p. 93°.

Lellmann, Mayer, *Ber.*, 1892, 25, 3582.

Dict. of Org. Comp.—III.

**3-Nitrobenzyl-m-toluidine**

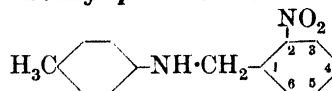


C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub> MW, 242

Cryst. from EtOH. M.p. 67°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, hot EtOH. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O, pet. ether.

Purgotti, Monti, *Gazz. chim. ital.*, 1900, 30, ii, 258.

**2-Nitrobenzyl-p-toluidine**



C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub> MW, 242

Yellow cryst. M.p. 72°. Sol. most org. solvents. Spar. sol. ligroin.

**N-Formyl**: needles from EtOH. M.p. 79°.

**N-Acetyl**: *N*-2-nitrobenzylacet-*p*-toluidide. Cryst. from CHCl<sub>3</sub>-Et<sub>2</sub>O. M.p. 65°.

**N-Benzenesulphonyl**: cryst. from EtOH. M.p. 124°.

**N-Nitroso**: yellow needles from EtOH. M.p. 80°.

Lellmann, Stickel, *Ber.*, 1886, 19, 1609.

Busch, *J. prakt. Chem.*, 1895, 51, 271.

**3-Nitrobenzyl-p-toluidine.**

Needles from EtOH. M.p. 86°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, hot EtOH. Spar. sol. pet. ether.

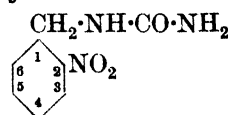
Purgotti, Monti, *Gazz. chim. ital.*, 1900, 30, ii, 258.

**4-Nitrobenzyl-p-toluidine.**

Yellow cryst. M.p. 68°.

Lellmann, Mayer, *Ber.*, 1892, 25, 3582.

**o-Nitrobenzylurea**



C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N<sub>3</sub> MW, 195

Needles from H<sub>2</sub>O. M.p. 150°.

Gabriel, Jansen, *Ber.*, 1891, 24, 3092.

**p-Nitrobenzylurea.**

Yellow needles from EtOH. M.p. 196-7°. Sol. EtOH, AcOH. Insol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Hafner, *Ber.*, 1890, 23, 339.

**Nitrobiuret**



C<sub>2</sub>H<sub>4</sub>O<sub>4</sub>N<sub>4</sub> MW, 148

Cryst. powder from  $\text{H}_2\text{O}$ . M.p.  $165^\circ$  ( $223^\circ$ ) decomp. Sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Aq. sol. reacts acid. Boiling  $\text{H}_2\text{O} \longrightarrow \text{urea} + \text{CO}_2 + \text{N}_2\text{O}$ . Does not give biuret reaction with  $\text{CuSO}_4 + \text{alkali}$ .

Thiele, Uhlfelder, *Ann.*, 1898, **303**, 95.

Davis, Blanchard, *J. Am. Chem. Soc.*, 1929, **51**, 1801.

### Nitrobromoform.

See Bromopiricin.

### 1-Nitrobutane



$\text{C}_4\text{H}_9\text{O}_2\text{N}$  MW, 103

Colourless liq. B.p.  $151-2^\circ$ . Sol. aq. alkalis.

Züblin, *Ber.*, 1877, **10**, 2083.

Rây, Neogi, *J. Chem. Soc.*, 1906, **89**, 1902.

### 2-Nitrobutane



$\text{C}_4\text{H}_9\text{O}_2\text{N}$  MW, 103

Liq. B.p.  $138-9^\circ/747$  mm.  $D_4^{20}$  0.9877.

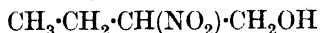
Meyer, Locher, *Ann.*, 1876, **180**, 134.

Bewad, *J. prakt. Chem.*, 1901, **63**, 194.

### Nitrobutanol.

See Nitrobutyl Alcohol.

### 2-Nitro-n-butyl Alcohol (2-Nitrobutanol-1)



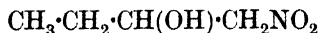
$\text{C}_4\text{H}_9\text{O}_3\text{N}$  MW, 119

Liq. B.p.  $127-30^\circ/25$  mm.  $D_4^{15}$  1.1365. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{AcOH}$ . Mod. sol.  $\text{H}_2\text{O}$ .

Henry, *Bull. soc. chim.*, 1896, **15**, 1223.

Pauwels, *Chem. Zentr.*, 1898, **I**, 193.

### 1-Nitro-sec.-n-butyl Alcohol (1-Nitrobutanol-2)



$\text{C}_4\text{H}_9\text{O}_3\text{N}$  MW, 119

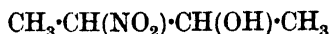
Thick liq. B.p.  $204^\circ/767$  mm., slight decomp.,  $123-5^\circ/35$  mm.  $D_4^{18}$  1.191,  $D_4^{25}$  1.144.

Acetyl: b.p.  $105-6^\circ/11$  mm.  $D_4^{20}$  1.1224.  $n_D^{20}$  1.4285.

Schmidt, Rutz, *Ber.*, 1928, **61**, 2142.

See also first reference above.

### 3-Nitro-sec.-n-butyl Alcohol (3-Nitrobutanol-2)



$\text{C}_4\text{H}_9\text{O}_3\text{N}$  MW, 119

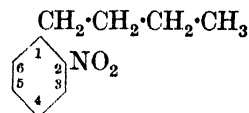
B.p.  $112-13^\circ/38$  mm.  $D_4^{18}$  1.116.

Henry, *Bull. soc. chim.*, 1896, **15**, 1224.

### N-Nitrobutylamine.

See Butylnitramine.

### o-Nitro-n-butylbenzene (1-o-Nitrophenyl-n-butane)



$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

B.p.  $260^\circ$  part decomp.,  $131-3^\circ/15$  mm.  $D_4^{20}$  1.071.  $\text{Sn} + \text{HCl} \longrightarrow$  o-amino-n-butylbenzene. Volatile in steam.

Read, Mullin, *J. Am. Chem. Soc.*, 1928, **50**, 1763.

Reilly, Hickinbottom, *J. Chem. Soc.*, 1920, **117**, 116.

### m-Nitro-n-butylbenzene (1-m-Nitrophenyl-n-butane).

B.p.  $275^\circ/752$  mm. Misc. with  $\text{CHCl}_3$ ,  $\text{PhNO}_2$ ,  $\text{Py}$ , pet, ether. Volatile in steam.

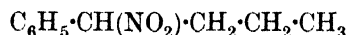
Reilly, Hickinbottom, *J. Chem. Soc.*, 1920, **117**, 118.

### p-Nitro-n-butylbenzene (1-p-Nitrophenyl-n-butane)

B.p.  $143-5^\circ/15$  mm.  $D_4^{20}$  1.065.  $\text{Sn} + \text{HCl} \longrightarrow$  p-amino-n-butylbenzene.

Read, Mullin, *J. Am. Chem. Soc.*, 1928, **50**, 1763.

### $\alpha$ -Nitro-n-butylbenzene (1-Nitro-1-phenyl-n-butane)

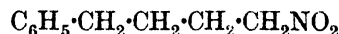


$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

B.p.  $250-6^\circ/758$  mm. decomp.,  $151-2^\circ/25$  mm.  $D_4^0$  1.0756,  $D_4^{20}$  1.0592.  $n_D^{20}$  1.50746.

Konowalow, *Ber.*, 1895, **28**, 1857.

### $\omega$ -Nitro-n-butylbenzene (4-Nitro-1-phenyl-n-butane)

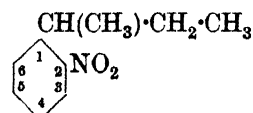


$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

B.p.  $160-5^\circ/15$  mm.

v. Braun, Kruber, *Ber.*, 1912, **45**, 397.

### o-Nitro-sec.-n-butylbenzene (2-o-Nitrophenyl-n-butane)



$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

B.p.  $123-6^\circ/12$  mm.  $D_4^{20}$  1.065.  $\text{Sn} + \text{HCl} \longrightarrow$  o-amino-sec.-n-butylbenzene.

Read, Hewitt, Pike, *J. Am. Chem. Soc.*, 1932, **54**, 1194.

**p-Nitro-sec.-n-butylbenzene** (2-p-Nitro-phenyl-n-butane).

B.p. 142-4°/12 mm. 130°/9 mm.  $D_4^{20}$  1.065.  
 $\text{Sn} + \text{HCl} \longrightarrow$  p-amino-sec.-n-butylbenzene.

See previous reference and also  
 Glattfeld, Wertheim, *J. Am. Chem. Soc.*,  
 1921, **43**, 2684.

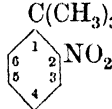
Harrison, Kenyon, Shepherd, *J. Chem. Soc.*, 1926, 660.

**$\omega$ -Nitro-sec.-n-butylbenzene** (4-Nitro-2-phenylbutane)

$\text{C}_6\text{H}_5 \cdot \overset{\text{CH}_3}{\underset{|}{\text{C}}} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{NO}_2$   
 $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179  
 B.p. 138°/12 mm.

v. Braun, Grabowski, Kirschbaum, *Ber.*,  
 1913, **46**, 1282.

**o-Nitro-tert.-butylbenzene**

$\text{C}(\text{CH}_3)_3$   
  
 $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179  
 Yellow liq. B.p. 250.5°/765 mm., 114.5°/10 mm.  $D^{16}$  1.074. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Volatile in steam.  $\text{Sn} + \text{HCl} \longrightarrow$  o-amino-tert.-butylbenzene.

Seńkowski, *Ber.*, 1890, **23**, 2414.  
 Shoesmith, Mackie, *J. Chem. Soc.*, 1928, 2334.

**m-Nitro-tert.-butylbenzene.**

Yellowish-red liq. M.p. below 20°. B.p. 250-2°/704 mm.

Gelzer, *Ber.*, 1888, **21**, 2946.  
 Seńkowski, *Ber.*, 1891, **24**, 2974.  
 Cf. Shoesmith, Mackie, *J. Chem. Soc.*, 1928, 2334.

**p-Nitro-tert.-butylbenzene.**

B.p. 265-7°/757 mm. slight decomp., 142-3°/17 mm., 125-30°/10 mm.  $\text{Sn} + \text{HCl} \longrightarrow$  p-amino-tert.-butylbenzene. Dil.  $\text{HNO}_3 \longrightarrow$  p-nitrobenzoic acid.

Malherbe, *Ber.*, 1919, **52**, 319.  
 Shoesmith, Mackie, *J. Chem. Soc.*, 1928, 2336.

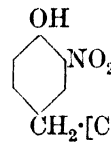
**$\omega$ -Nitro-tert.-butylbenzene**

$\text{C}_6\text{H}_5 \cdot \overset{\text{CH}_3}{\underset{\text{CH}_3}{\underset{|}{\text{C}}}} \cdot \text{CH}_2 \cdot \text{NO}_2$   
 $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

Yellow oil. B.p. 141-3°/15 mm.  $D_0^0$  1.0993,  $D_0^{20}$  1.0840.  $n_D^{27}$  1.52138. Sol. alkalis.

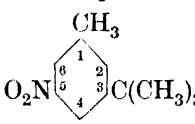
Konowalow, *Jahresber. Fortschr. Chem.*, 1895, 1538.

**2-Nitro-4-butylphenol**

  
 $\text{C}_{10}\text{H}_{13}\text{O}_3\text{N}$  MW, 195  
 B.p. 125°/3.5 mm.

Baranger, *Bull. soc. chim.*, 1931, **49**, 1213.

**5-Nitro-3-tert.-butyltoluene**

$\text{CH}_3$   
  
 $\text{C}_{11}\text{H}_{15}\text{O}_2\text{N}$  MW, 193  
 M.p. 32°. B.p. 120°/15 mm.

Baur-Thurgau, *Ber.*, 1897, **30**, 303.

**6-Nitro-3-tert.-butyltoluene.**

Liq. B.p. 160-2° in vacuo. Volatile in steam.

Baur, *Ber.*, 1891, **24**, 2835.

**2-Nitro-4-tert.-butyltoluene.**

Yellow oil. B.p. 138-9°.

Battegay, Haefely, *Bull. soc. chim.*, 1924, **35**, 981.

**1-Nitrobutyric Acid**

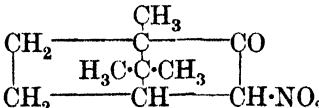
$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}(\text{NO}_2) \cdot \text{COOH}$   
 $\text{C}_4\text{H}_7\text{O}_4\text{N}$  MW, 133  
*Et ester*:  $\text{C}_6\text{H}_{11}\text{O}_4\text{N}$ . MW, 161. Yellow oil. B.p. 123°/20 mm.  $n_D^{18}$  1.4535. Spar. sol. H<sub>2</sub>O. Easily sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Schmidt, Widmann, *Ber.*, 1909, **42**, 1896.

**Nitrocaffeic Acid.**

See Nitro-3:4-dihydroxycinnamic Acid.

**3-Nitro-d-camphor** ( $\alpha$ -Nitro-d-camphor)

  
 $\text{C}_{10}\text{H}_{15}\text{O}_3\text{N}$  MW, 197  
*Nitro-form.*

Prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 103° (100-1°). Very sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Sol. EtOH, Et<sub>2</sub>O, ligroin. Spar. sol. pet. ether. Shows mutarotation.  $[\alpha]_D^{15} - 124^\circ \longrightarrow -104^\circ$  in C<sub>6</sub>H<sub>6</sub>.  $[\alpha]_D^{18} - 27^\circ \longrightarrow -15^\circ$  in CHCl<sub>3</sub>.  $[\alpha]_D^{18} - 26^\circ \longrightarrow -9^\circ$  in EtOH.  $[\alpha]_D^{18} - 3^\circ \longrightarrow +8^\circ$  in AcOH.

*Acti-form.* 3-Isonitro-*d*-camphor.

Not obtained pure in free state.

*NH<sub>4</sub> salt*: cryst. from EtOH. M.p. 178°.  $[\alpha]_{546}^{20} + 384^\circ$  in H<sub>2</sub>O. Sol. H<sub>2</sub>O. Mod. sol. EtOH. Spar. sol. Me<sub>2</sub>CO. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether.

*Na salt*: cryst. from EtOH. Very sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

*Cu salt*: green cryst. from EtOH. Sol. EtOH, Et<sub>2</sub>O with brown col. Insol. H<sub>2</sub>O.

*Anhydride*: C<sub>20</sub>O<sub>28</sub>O<sub>5</sub>N<sub>2</sub>. MW, 376. Exists in two forms. (i) Cryst. from EtOH. M.p. 193°.  $[\alpha]_{546}^{20} + 242^\circ$  in C<sub>6</sub>H<sub>6</sub>. (ii) Greenish-yellow needles from EtOH. M.p. 184°.  $[\alpha]_{546}^{20} - 6^\circ$  in C<sub>6</sub>H<sub>6</sub>.

Lowry, Steele, *J. Chem. Soc.*, 1915, 107, 1040.

Lowry, *J. Chem. Soc.*, 1898, 73, 995.

Forster, *J. Chem. Soc.*, 1902, 81, 868.

Cazeneuve, *Bull. soc. chim.*, 1888, 49, 92.

Lowry, Robertson, *J. Chem. Soc.*, 1904, 85, 1545.

### N-Nitrocarbamic Acid



CH<sub>2</sub>O<sub>4</sub>N<sub>2</sub> MW, 106

*K salt*: needles. Decomp. by H<sub>2</sub>O. Sol. very conc. KOH. Explodes on heating.

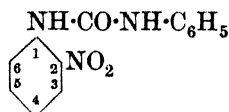
*Me ester*: C<sub>2</sub>H<sub>4</sub>O<sub>4</sub>N<sub>2</sub>. MW, 120. Plates or prisms. M.p. 88°. Decomp. at 120–30°.

*Et ester*: see Nitrourethane.

*Amide*: see Nitrourea.

Thiele, Dent, *Ann.*, 1898, 302, 249.

### 2-Nitrocarbanilide



C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>N<sub>3</sub> MW, 257

Yellowish needles from H<sub>2</sub>O or EtOH. M.p. 231–3° (170°). Sol. hot EtOH. Insol. Et<sub>2</sub>O, pet. ether.

Manuelli, Comanducci, *Gazz. chim. ital.*, 1899, 29, ii, 141.

### 3-Nitrocarbanilide.

Pale yellow needles. M.p. 198.5°.

Leuckart, *J. prakt. Chem.*, 1890, 41, 322.

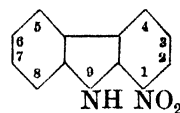
See also previous reference.

### 4-Nitrocarbanilide.

Cryst. from EtOH.Aq. M.p. 212° (202°).

See previous references.

### 1-Nitrocarbazole



C<sub>12</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub> MW, 212

Yellow needles from AcOH. M.p. 187°. Mod. sol. CS<sub>2</sub>. Spar. sol. pet. ether. Alc. KOH → bluish-red sol.

Lindemann, *Ber.*, 1924, 57, 555.

I.G., D.R.P., 511,021, (*Chem. Abstracts*, 1931, 25, 1262).

### 3-Nitrocarbazole.

Cryst. M.p. 214° (205°). Spar. sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOH. Insol. Et<sub>2</sub>O, pet. ether.

*N-Acetyl*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 237–8°. Sol. CHCl<sub>3</sub>. Spar. sol. EtOH. Almost insol. pet. ether.

*N-Benzoyl*: yellow plates from AcOH. M.p. 181°. Sol. Et<sub>2</sub>O, warm C<sub>6</sub>H<sub>6</sub>, ligroin.

*N-Nitroso*: pale yellow needles from EtOH. M.p. 166.5° decomp. Sol. hot AcOEt. Mod. sol. CHCl<sub>3</sub>. Spar. sol. ligroin.

*N-Me*: C<sub>13</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>. MW, 226. Yellow needles from EtOH. M.p. 173° (169–71°).

*N-Et*: C<sub>14</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 240. Yellow needles from EtOH. M.p. 126–8°.

Lindemann, *Ber.*, 1924, 57, 555.

Mazzara, *Ber.*, 1891, 24, 281.

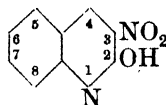
Mazzara, Leonardi, *Gazz. chim. ital.*, 1892, 22, ii, 443.

Stevens, Tucker, *J. Chem. Soc.*, 1923, 123, 2143.

### 3-Nitrocarbostyryl

(3-Nitro-2-hydroxy-

quinoline)



C<sub>9</sub>H<sub>6</sub>O<sub>3</sub>N<sub>2</sub> MW, 190

Fine needles from EtOH. Does not melt below 320°. Spar. sol. EtOH, Me<sub>2</sub>CO, AcOH.

Friedländer, Lazarus, *Ann.*, 1885, 229, 243.

### 4-Nitrocarbostyryl

(4-Nitro-2-hydroxy-

quinoline).

Yellow needles from AcOH. M.p. 260°. Insol. EtOH. Sol. dil. alkalis.

See previous reference.

### 5-Nitrocarbostyryl

(5-Nitro-2-hydroxy-

quinoline).

Golden-yellow needles or leaflets from EtOH. M.p. 304°. Very spar. sol. EtOH.

*Me ether*:  $C_{10}H_8O_3N_2$ . MW, 204. Yellow needles. M.p.  $151^\circ$ . Sol. EtOH. Insol.  $H_2O$ .

*N-Me*: 5-nitro-*N*-methyl- $\alpha$ -quinolone.  $C_{10}H_8O_3N_2$ . MW, 204. Yellow needles from EtOH or  $H_2O$ . M.p.  $167^\circ$ . Sol. Et<sub>2</sub>O,  $C_6H_6$ . Sublimes.

*N-Et*: 5-nitro-*N*-ethyl- $\alpha$ -quinolone.  $C_{11}H_{10}O_3N_2$ . MW, 218. Cryst. from MeOH. M.p.  $135^\circ$ . Sol. EtOH,  $C_6H_6$ , hot  $H_2O$ .

Claus, Setzer, *J. prakt. Chem.*, 1896, **53**, 392.

Fischer, Guthmann, *J. prakt. Chem.*, 1916, **93**, 383.

Decker, Remfry, *Ber.*, 1905, **38**, 2776.

**6-Nitrocarbostyryl** (6-Nitro-2-hydroxy-quinoline).

Yellowish needles from  $H_2O$ . M.p.  $283^\circ$  ( $277^\circ$ ).

*Me ether*:  $C_{10}H_8O_3N_2$ . MW, 204. Needles from  $C_6H_6$ . M.p.  $189-90^\circ$ . Very sol.  $C_6H_6$ ,  $CHCl_3$ , toluene. Sol. Et<sub>2</sub>O, EtOH, ligroin. Insol.  $H_2O$ .

*Et ether*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Needles from  $C_6H_6$ . M.p.  $156-8^\circ$ .

*N-Me*: 6-nitro-*N*-methyl- $\alpha$ -quinolone. Yellow needles from EtOH. M.p.  $226^\circ$  ( $222^\circ$ ). Sublimes with slight decomp.

*N-Et*: 6-nitro-*N*-ethyl- $\alpha$ -quinolone. Yellow needles from EtOH. M.p.  $183^\circ$ . Sublimes.

Decker, *J. prakt. Chem.*, 1901, **64**, 89.

Fischer, Guthmann, *J. prakt. Chem.*, 1916, **93**, 378.

Kaufmann, de Petherd, *Ber.*, 1917, **50**, 343.

**7-Nitrocarbostyryl** (7-Nitro-2-hydroxy-quinoline).

Pale yellow needles from 1000 parts boiling amyl alcohol. M.p. about  $340^\circ$ . Spar. sol. most org. solvents.

*N-Me*: 7-nitro-*N*-methyl- $\alpha$ -quinolone. Yellow needles from EtOH. M.p.  $198-9^\circ$ . Sol.  $C_6H_6$ .

*N-Et*: 7-nitro-*N*-ethyl- $\alpha$ -quinolone. Needles from EtOH. M.p.  $168-9^\circ$ .

Decker, *J. prakt. Chem.*, 1901, **64**, 99.

**8-Nitrocarbostyryl** (8-Nitro-2-hydroxy-quinoline).

Yellow prisms from  $H_2O$ . M.p.  $168^\circ$  ( $163^\circ$ ). Spar. sol. cold EtOH.

*B.HCl*: m.p.  $159-60^\circ$ .

*N-Me*: 8-nitro-*N*-methyl- $\alpha$ -quinolone. Yellow needles from  $C_6H_6$ . M.p.  $133-4^\circ$  ( $128^\circ$  from EtOH).

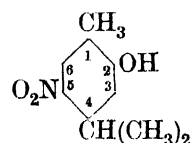
*N-Et*: 8-nitro-*N*-ethyl- $\alpha$ -quinolone. Yellow cryst. M.p.  $96^\circ$ . Very sol.  $C_6H_6$ .

Decker, Pollitz, *J. prakt. Chem.*, 1901, **64**, 91.

Fischer, Guthmann, *J. prakt. Chem.*, 1916, **93**, 378.

Decker, *Ber.*, 1905, **38**, 1151.

**5-Nitrocarvacrol** (5-Nitro-2-hydroxy-*p*-cymene)



$C_{10}H_{13}O_3N$  MW, 195

Pale yellow prisms from  $C_6H_6$ -ligroin. M.p.  $87^\circ$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol.  $H_2O$ .

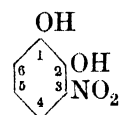
Kehrmann, Schön, *Ann.*, 1900, **310**, 109.

**6-Nitrocarvacrol** (6-Nitro-2-hydroxy-*p*-cymene).

Needles. M.p.  $116-17^\circ$ .

Aschan, *Chem. Zentr.*, 1919, **I**, 227.

**3-Nitrocatechol**



$C_6H_5O_4N$  MW, 155

Yellow needles from pet. ether. M.p.  $86.5^\circ$ . NaOH  $\rightarrow$  reddish-purple sol.

*1-Me ether*: 3-nitroguaiacol.  $C_7H_7O_4N$ . MW, 169. Yellow needles. M.p.  $62^\circ$ . Sol.  $H_2O$ . Spar. sol. pet. ether. Sublimes. *2-Acetyl*: leaflets from pet. ether. M.p.  $40^\circ$ . Sol. usual org. solvents. *2-Benzoyl*: plates from EtOH. M.p.  $88-9^\circ$ .

*2-Me ether*: 6-nitroguaiacol. M.p.  $102-3^\circ$ .

*Di-Me ether*: 3-nitroveratrol.  $C_8H_9O_4N$ . MW, 183. Needles from EtOH. M.p.  $64-5^\circ$ . Sol. EtOH, Et<sub>2</sub>O,  $C_6H_6$ . Insol.  $H_2O$ , ligroin.

Vermeulen, *Rec. trav. chim.*, 1906, **25**, 23.

Klemenc, *Monatsh.*, 1912, **33**, 704.

Berkenheim, Albitzkaja, *Chem. Zentr.*, 1935, **I**, 2794.

**4-Nitrocatechol**.

Yellow needles from  $H_2O$ . M.p.  $176^\circ$ . Sol.  $H_2O$ , EtOH, Et<sub>2</sub>O. Spar. sol.  $C_6H_6$ . KOH  $\rightarrow$  reddish-purple sol.

*1-Me ether*: 4-nitroguaiacol.  $C_7H_7O_4N$ . MW, 169. Pale yellow needles from  $H_2O$ . M.p.  $105^\circ$ . Dil. KOH  $\rightarrow$  orange sol. *2-Et ether*:  $C_9H_{11}O_4N$ . MW, 197. Cryst. from EtOH.

M.p. 100–2° 2-Acetyl: needles from hot H<sub>2</sub>O. M.p. 101–2°. Sol. boiling EtOH. Insol. cold H<sub>2</sub>O.

2-Me ether: 5-nitroguaiacol. Yellow needles from H<sub>2</sub>O. M.p. 103–4°. Sol. EtOH, Et<sub>2</sub>O, boiling H<sub>2</sub>O. Dil. KOH → orange-yellow sol.

1-Et ether: M.p. 85–6° 1-Acetyl: needles from H<sub>2</sub>O. M.p. 108–9°. Sol. H<sub>2</sub>O.

Di-Me ether: 4-nitroveratrol. C<sub>8</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 183. Yellow needles from EtOH. Aq. M.p. 96°. B.p. about 230°/15–20 mm. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, ligroin.

1-Et ether: C<sub>8</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 183. Pale yellow prisms from MeOH. Aq. M.p. 113–14°. Benzoyl: needles from MeOH. M.p. 101–2°.

Di-Et ether: C<sub>10</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 211. Pale yellow needles. M.p. 73–5°.

Diacetyl: m.p. 98°.

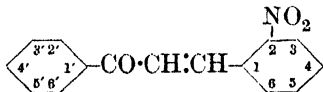
Dibenzoyl: needles from EtOH. M.p. 156°. Spar. sol. MeOH.

Cardwell, Robinson, *J. Chem. Soc.*, 1915, 107, 258.

Pollecoff, Robinson, *J. Chem. Soc.*, 1918, 113, 647.

Riedel, D.R.P., 264,012, (*Chem. Zentr.*, 1913, II, 1181).

**2-Nitrochalkone** (ω-2-Nitrobenzylideneacetophenone)



C<sub>15</sub>H<sub>11</sub>O<sub>3</sub>N MW, 253

Pale brown needles from EtOH. M.p. 125°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with greenish-red fluor.

Semicarbazone: golden-yellow leaflets from EtOH. M.p. 177–5°.

Phenylhydrazone: cryst. from EtOH. M.p. 162–4°.

Sorge, *Ber.*, 1902, 35, 1067.

Tanasescu, Georgescu, *J. prakt. Chem.*, 1934, 139, 189.

### 3-Nitrochalkone.

Yellow needles from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 145–6°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, CHCl<sub>3</sub>, AcOH. Insol. Et<sub>2</sub>O, ligroin.

Phenylhydrazone: yellow cryst. from EtOH. M.p. 101–3°.

Ruhemann, *J. Chem. Soc.*, 1903, 83, 1377. See also previous references.

### 4-Nitrochalkone.

Pale yellow needles from EtOH, plates from C<sub>6</sub>H<sub>6</sub>. M.p. 164°. Sol. CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O, ligroin.

Semicarbazone: cryst. from EtOH. M.p. 178–9°.

Phenylhydrazone: red needles from EtOH. M.p. 148–50°.

Sorge, *Ber.*, 1902, 35, 1068.

Tanasescu, Georgescu, *J. prakt. Chem.*, 1934, 139, 189.

### 2'-Nitrochalkone.

Needles from EtOH. M.p. 128–9° (124°). Conc. H<sub>2</sub>SO<sub>4</sub> → pale yellow sol.

Hydrazone: m.p. 146–7°.

Engler, Dorant, *Ber.*, 1895, 28, 2498.

### 3'-Nitrochalkone.

Cryst. from EtOH. M.p. 131°. Conc. H<sub>2</sub>SO<sub>4</sub> → orange-yellow sol.

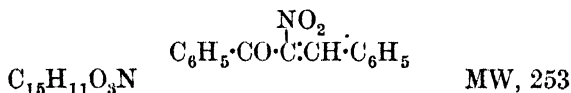
Dilthey, Neuhaus, Schommer, *J. prakt. Chem.*, 1929, 123, 235.

### 4'-Nitrochalkone.

Yellowish cryst. from EtOH. M.p. 149–50°. Conc. H<sub>2</sub>SO<sub>4</sub> → yellowish-orange sol.

See previous reference.

**β-Nitrochalkone** (α-Nitrochalkone, ω-nitro-ω-benzylideneacetophenone)



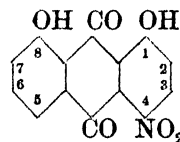
Yellow cryst. from AcOH, plates from Et<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>. M.p. 90°. Sol. most org. solvents.

Wieland, *Ann.*, 1903, 328, 237.

### Nitrochloroform.

See Chloropicrin.

**4-Nitrochrysazin** (4-Nitro-1:8-dihydroxy-anthraquinone)

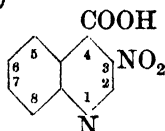


C<sub>14</sub>H<sub>7</sub>O<sub>6</sub>N MW, 285

Orange-yellow cryst. from chlorobenzene. M.p. 232–4°. Alkalis give red sols. Conc. H<sub>2</sub>SO<sub>4</sub> → orange-yellow sol.

Di-Me ether: C<sub>16</sub>H<sub>11</sub>O<sub>6</sub>N. MW, 313. Greenish-yellow needles from chlorobenzene. M.p. 232–3°. Spar. sol. org. solvents. Insol. H<sub>2</sub>O, alkalis. Conc. H<sub>2</sub>SO<sub>4</sub> → orange-red sol.

M.L.B., D.R.P., 193,104, (*Chem. Zentr.*, 1908, I, 428).

**3-Nitrocinchoninic Acid** (3-Nitroquinoline-4-carboxylic acid) $C_{10}H_6O_4N_2$ 

MW, 218

Needles from EtOH. M.p. 204°.

Badische, D.R.P., 335,197, (*Chem. Zentr.*, 1921, II, 962).**5-Nitrocinchoninic Acid.**

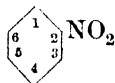
Yellowish cryst. powder. M.p. 275-8° decomp. Sol. hot AcOH, conc. HCl. Spar. sol. other solvents.

Königs, Lossow, *Ber.*, 1899, 32, 717.**6-Nitrocinchoninic Acid.**

Cryst. M.p. above 280° decomp.

Strache, *Monatsh.*, 1889, 10, 645.**o-Nitrocinnamaldehyde**

CH:CH-CHO

 $C_9H_7O_3N$ 

MW, 177

Needles from Et<sub>2</sub>O or EtOH. M.p. 127-127.5°. Sol. hot H<sub>2</sub>O, CHCl<sub>3</sub>. Less sol. EtOH, Et<sub>2</sub>O.

anti-Oxime: pearly leaflets from EtOH. M.p. 134°. Acetyl: m.p. 107°.

syn-Oxime: cryst. from Me<sub>2</sub>CO.Aq. M.p. 140°. B, HCl: m.p. 144°.Mills, Evans, *J. Chem. Soc.*, 1920, 117, 1037.Brady, Grayson, *J. Chem. Soc.*, 1924, 125, 1421.**m-Nitrocinnamaldehyde.**Needles from H<sub>2</sub>O, prisms from EtOH. M.p. 116°. Sol. C<sub>6</sub>H<sub>6</sub>, AcOH. Spar. sol. H<sub>2</sub>O, cold EtOH, Et<sub>2</sub>O.

anti-Oxime: yellow plates from EtOH. M.p. 157°. Acetyl: m.p. 136°.

syn-Oxime: yellow cryst. powder from Me<sub>2</sub>CO.Aq. M.p. 163°. B, HCl: m.p. 175°.

Phenylhydrazone: red plates from EtOH. M.p. 160°.

Kinkelin, *Ber.*, 1885, 18, 484.Brady, Grayson, *J. Chem. Soc.*, 1924, 125, 1420.**p-Nitrocinnamaldehyde.**Needles from H<sub>2</sub>O or EtOH. M.p. 141-2°. Sol. usual solvents.

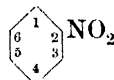
anti-Oxime: yellow cryst. from EtOH. M.p. 178-9°. Acetyl: yellow cryst. M.p. 158°.

Phenylhydrazone: orange-red cryst. from EtOH. M.p. 180-1°.

Anil: needles from EtOH. M.p. 132-3°.

Einhorn, Gehrenbeck, *Ann.*, 1889, 253, 348.Fecht, *Ber.*, 1907, 40, 3898.**o-Nitrocinnamic Acid**

CH:CH-COOH

 $C_9H_7O_4N$ 

MW, 193

*Cis*:Yellowish cryst. from C<sub>6</sub>H<sub>6</sub> or CHCl<sub>3</sub>. M.p. 146-7° (143°). Sol. EtOH, hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. CHCl<sub>3</sub>. Warm conc. H<sub>2</sub>SO<sub>4</sub> → dark blue sol. Bromine in sunlight → *trans* form.*Trans*:Needles from EtOH. M.p. 240°. Sol. boiling EtOH. Insol. H<sub>2</sub>O. Turns red in sunlight. Ultraviolet absorption in Py.Aq. → 22% *cis* form.*Me ester*: C<sub>10</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 207. Needles from H<sub>2</sub>O. M.p. 73°. B.p. 187-9°/15 mm. Very sol. boiling EtOH. Spar. sol. boiling H<sub>2</sub>O.*Et ester*: C<sub>11</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 221. Yellow cryst. from EtOH. M.p. 44°. Very sol. warm EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>.*Menthyl ester*: cryst. from 95% EtOH. M.p. 49.5°.*Cyclohexyl ester*: m.p. 55-6°.*p-Nitrobenzyl ester*: cryst. from EtOH. M.p. 132°.*Phenacyl ester*: m.p. 126°.*Chloride*: C<sub>9</sub>H<sub>6</sub>O<sub>3</sub>NCl. MW, 211.5. Cryst. M.p. 64.5°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.*Amide*: C<sub>9</sub>H<sub>6</sub>O<sub>3</sub>N<sub>2</sub>. MW, 192. Needles from H<sub>2</sub>O. M.p. 185°. Sol. 100 parts H<sub>2</sub>O. Sol. hot EtOH, Me<sub>2</sub>CO, AcOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOEt. Insol. ligroin.*Chloroamide*: C<sub>9</sub>H<sub>7</sub>O<sub>3</sub>N<sub>2</sub>Cl. MW, 226.5. Needles from AcOH. M.p. 142°. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, ligroin.*Nitrile*: C<sub>9</sub>H<sub>6</sub>O<sub>2</sub>N<sub>2</sub>. MW, 174. Needles from H<sub>2</sub>O. M.p. 92°. B.p. 194-6°/7-8 mm. Sol. about 460 parts H<sub>2</sub>O. Spar. sol. ligroin.Tanasescu, *Bull. soc. chim.*, 1927, 41, 1074. Weerman, *Ann.*, 1913, 401, 9.Stoermer, Heymann, *Ber.*, 1912, 45, 3100. Pschorr, *Ber.*, 1898, 31, 1295.Sudborough, Lloyd, *J. Chem. Soc.*, 1898, 73, 85.



**m-Nitrocinnamic Acid.**

*Cis* :

Needles. M.p. 158°. Bromine in sunlight → *trans* form.

*Trans* :

Needles from EtOH. M.p. 200–1°. Sol. 100 parts EtOH. Ultraviolet absorption in EtOH·NH<sub>3</sub>·Aq. → 22% *cis* form.

*Me ester* : C<sub>10</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 207. Pale yellow prisms from MeOH. M.p. 123–4°. Sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. MeOH, EtOH, Et<sub>2</sub>O, CS<sub>2</sub>.

*Et ester* : C<sub>11</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 221. Prisms from AcOH. M.p. 78–9°.

*Menthyl ester* : m.p. 85°.

*p-Nitrobenzyl ester* : cryst. from EtOH. M.p. 173·5–174°.

*Phenacyl ester* : m.p. 145·5°.

*p-Bromophenacyl ester* : m.p. 178°.

*Amide* : C<sub>9</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>. MW, 192. Leaflets from MeOH. M.p. 195–6°. Sol. EtOH, hot H<sub>2</sub>O, AcOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, ligroin.

*Chloroamide* : C<sub>9</sub>H<sub>7</sub>O<sub>3</sub>N<sub>2</sub>Cl. MW, 226·5. Cryst. from AcOH. M.p. 178° decomp.

*Hydrazide* : pale yellow cryst. from EtOH. M.p. 139°. *B,HCl* : m.p. 212° decomp. *Benzoyl* : m.p. 185·5°.

*Dihydrazide* : pale yellow cryst. from EtOH. M.p. 279° decomp. *B,HCl* : m.p. 256° decomp.

*Azide* : m.p. 117–18°.

Wollring, *Ber.*, 1914, **47**, 112.

Weerman, *Ann.*, 1913, **401**, 15.

Sudborough, Lloyd, *J. Chem. Soc.*, 1898, **73**, 85.

Thayer, *Organic Syntheses*, Collective Vol. I, 390.

**p-Nitrocinnamic Acid.**

*Cis* :

Yellow cryst. from toluene or CHCl<sub>3</sub>. M.p. 143°. Sol. C<sub>6</sub>H<sub>6</sub>, EtOH. Bromine → *trans* form.

*Trans* :

Prisms from EtOH. M.p. 286°. Spar. sol. boiling H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Insol. CS<sub>2</sub>, pet. ether.

*Me ester* : C<sub>10</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 207. Needles from EtOH. M.p. 161°. B.p. 281–6°. Spar. sol. EtOH. Insol. Et<sub>2</sub>O.

*Et ester* : C<sub>11</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 221. Plates from AcOH. M.p. 141–2°. Insol. cold EtOH. Spar. volatile in steam.

*Menthyl ester* : m.p. 92·5°.

*Phenyl ester* : C<sub>15</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 269. Needles from EtOH. M.p. 152°.

*p-Nitrobenzyl ester* : cryst. from EtOH. M.p. 186·5°.

*Phenacyl ester* : m.p. 146·2°.

*p-Bromophenacyl ester* : m.p. 191°.

*Chloride* : C<sub>9</sub>H<sub>8</sub>O<sub>3</sub>NCl. MW, 211·5. Cryst. M.p. 124·5°. B.p. 205–10°.

*Amide* : C<sub>9</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>. MW, 192. Needles from EtOH. M.p. 217°. Spar. sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Chloroamide* : C<sub>9</sub>H<sub>7</sub>O<sub>3</sub>N<sub>2</sub>Cl. MW, 226·5. Needles from AcOH. M.p. 169° decomp.

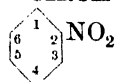
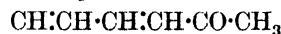
Wollring, *Ber.*, 1914, **47**, 112.

Pfeiffer, Haefelin, *Ber.*, 1922, **55**, 1771.

Weerman, *Ann.*, 1913 **401**, 18.

Beilstein, Kuhlberg, *Ann.*, 1872, **163**, 126.

**o-Nitrocinnamylideneacetone**



C<sub>12</sub>H<sub>11</sub>O<sub>3</sub>N MW, 217

Needles. M.p. 73·5°. Sol. usual solvents. Conc. H<sub>2</sub>SO<sub>4</sub> → orange-yellow sol.

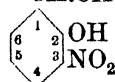
Diell, Einhorn, *Ber.*, 1885, **18**, 2327.

**p-Nitrocinnamylideneacetone.**

Needles from EtOH. M.p. 132°. Sol. usual solvents.

Einhorn, Gehrenbeck, *Ann.*, 1889, **253**, 353.

**3-Nitro-o-coumaraldehyde**



C<sub>9</sub>H<sub>7</sub>O<sub>4</sub>N MW, 193

Golden-yellow needles from AcOH·Aq. M.p. 133°. Sol. hot H<sub>2</sub>O. Mod. sol. EtOH, AcOH. Less sol. Et<sub>2</sub>O.

*Me ether* : C<sub>10</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 207. Yellow prisms from EtOH. M.p. 115°.

v. Miller, Kinkelin, *Ber.*, 1887, **20**, 1933.

**5-Nitro-o-coumaraldehyde.**

Yellow needles from H<sub>2</sub>O. M.p. 200° decomp. Sol. EtOH, AcOH. Spar. sol. H<sub>2</sub>O.

See previous reference.

**3-Nitro-o-coumaric Acid**



C<sub>9</sub>H<sub>7</sub>O<sub>5</sub>N

MW, 209

*Cis*: 3-Nitrocoumarinic acid.

Yellow prisms from EtOH. M.p. 150° decomp. (rapid heat.). Warm H<sub>2</sub>O or EtOH → 8-nitrocoumarin.

*Me ether*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Leaflets. M.p. 135–6°. *Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Prisms from EtOH. M.p. 69°.

*Trans*:

Yellow cryst. from EtOH. M.p. 241–2°. Spar. sol. EtOH.

*Me ether*: yellow prisms from EtOH. M.p. 193°. *Me ester*: needles from EtOH. M.p. 88–9°.

v. Miller, Kinkelin, *Ber.*, 1889, 22, 1706.

### 5-Nitro-*o*-coumaric Acid.

*Cis*: 5-Nitrocoumarinic acid.

*Me ether*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Needles from EtOH or AcOH. Aq. M.p. 202–3°. *Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Needles from EtOH. M.p. 124–5°. *Et ester*: C<sub>12</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 251. Needles from EtOH. M.p. 75–7°.

*Et ether*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Needles. M.p. 171–2°. *Me ester*: C<sub>12</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 251. Needles from EtOH. M.p. 111–13°. *Et ester*: C<sub>13</sub>H<sub>15</sub>O<sub>5</sub>N. MW, 265. Needles from EtOH. M.p. 104–5°.

*Trans*:

Needles. M.p. 247° decomp. Turns yellow in air.

*Me ether*: needles from H<sub>2</sub>O. M.p. 239°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Needles from EtOH. M.p. 163°. *Et ester*: needles from EtOH. M.p. 85°.

*Et ether*: needles from EtOH. Aq. M.p. 194–5°. *Me ester*: needles from EtOH. M.p. 141–2°.

*Me ester*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Needles. M.p. 211°.

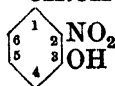
*Acetyl*: needles from hot AcOH. M.p. 217°, solidifies and remelts at 259°.

Clayton, *J. Chem. Soc.*, 1910, 97, 2106.

Dey, Row, *J. Chem. Soc.*, 1924, 125, 563.

### 2-Nitro-*m*-coumaric Acid

CH:CH·COOH



C<sub>9</sub>H<sub>7</sub>O<sub>5</sub>N

MW, 209

Needles from H<sub>2</sub>O. M.p. 218°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. CHCl<sub>3</sub>, pet. ether. Sweet taste.

Luff, *Ber.*, 1889, 22, 292.

### 4-Nitro-*m*-coumaric Acid.

Golden-yellow needles from EtOH. M.p. 248°. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. H<sub>2</sub>O.

*Me ether*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Needles from EtOH. M.p. 218° decomp. *Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Needles from EtOH. M.p. 163° (143°). Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Mod. sol. CHCl<sub>3</sub>.

Ulrich, *Ber.*, 1885, 18, 2572.

See also previous reference.

### 5-Nitro-*m*-coumaric Acid.

Cryst. Sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, AcOH. Decomp. on heating.

Luff, *Ber.*, 1889, 22, 295.

### 6-Nitro-*m*-coumaric Acid.

Needles from EtOH. M.p. 216°. Very sol. EtOH. Sol. hot H<sub>2</sub>O, Et<sub>2</sub>O, AcOH.

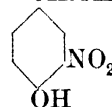
*Me ether*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Needles from EtOH. M.p. 224·5–225·5°. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Insol. ligroin. *Et ester*: C<sub>12</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 251. Needles from EtOH. M.p. 72·5°. Spar. sol. Et<sub>2</sub>O, ligroin.

Eichengrün, Einhorn, *Ann.*, 1891, 262, 171.

See also previous reference.

### 3-Nitro-*p*-coumaric Acid

CH:CH·COOH



C<sub>9</sub>H<sub>7</sub>O<sub>5</sub>N

MW, 209

Yellow needles from EtOH. M.p. 223° (198°) decomp. Insol. cold H<sub>2</sub>O.

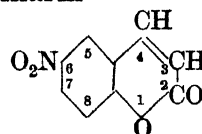
*Me ether*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Prisms or plates from EtOH, needles from H<sub>2</sub>O. M.p. 247–8°. Spar. sol. hot H<sub>2</sub>O, Et<sub>2</sub>O, CHCl<sub>3</sub>. *Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Cryst. from EtOH. M.p. 125°. *Et ester*: C<sub>12</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 251. Prisms from EtOH. M.p. 99–100°.

*Me ester*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Yellow needles from EtOH. M.p. 142–4°.

*Et ester*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Yellow needles from EtOH. M.p. 110–11°.

Johnson, Kohmann, *J. Am. Chem. Soc.*, 1915, 37, 165.

### 6-Nitrocoumarin



C<sub>9</sub>H<sub>5</sub>O<sub>4</sub>N

MW, 191

Needles. M.p. 185°. Mod. sol. boiling H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

*Oxime*: pale yellow needles. M.p. 249–50°.

*Phenylhydrazone*: red needles from AcOH. M.p. 213°.

Clayton, *J. Chem. Soc.*, 1910, **97**, 2106.

Clayton, Godden, *J. Chem. Soc.*, 1912, **101**, 213.

### 8-Nitrocoumarin.

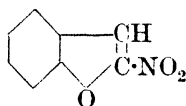
Prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 191°.

See previous references.

### Nitrocoumarinic Acid.

See under Nitro-*o*-coumaric Acid.

### 2-Nitrocoumarone



C<sub>8</sub>H<sub>5</sub>O<sub>3</sub>N

MW, 163

Needles. M.p. 134°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Mod. sol. MeOH, EtOH. Spar. sol. ligroin.

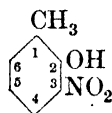
Stoermer, Richter, *Ber.*, 1897, **30**, 2094.

Stoermer, Kahlert, *Ber.*, 1902, **35**, 1641.

### 6-Nitrocresidine.

See under 6-Nitro-3-amino-*p*-cresol.

### 3-Nitro-*o*-cresol



C<sub>7</sub>H<sub>7</sub>O<sub>3</sub>N

MW, 153

Prisms from EtOH.Aq. or pet. ether. M.p. 70°. B.p. 102–3°/9 mm. Very sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Me ether*: C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 167. Needles from pet. ether. M.p. 30°. B.p. 121–2°/10 mm.

*Et ether*: C<sub>9</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 181. Yellow oil. B.p. 249–50°.

*Propyl ether*: C<sub>10</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 195. Yellow oil. B.p. 210–12°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Benzoyl*: needles from EtOH or pet. ether. M.p. 42°. B.p. 218–20°/9 mm.

*p*-Toluenesulphonyl: cryst. from EtOH. M.p. 66°. B.p. 205–10°/0.5 mm. Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, CCl<sub>4</sub>. Spar. sol. EtOH.

Gibson, *J. Chem. Soc.*, 1925, **127**, 44.

Spiegel, Munblit, Kaufmann, *Ber.*, 1906, **39**, 3242.

### 4-Nitro-*o*-cresol.

Yellow needles from ligroin. M.p. 118°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, CS<sub>2</sub>, ligroin.

*Me ether*: needles from EtOH or C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 74°. Spar. sol. EtOH, Et<sub>2</sub>O, AcOH. Insol. H<sub>2</sub>O.

*Et ether*: needles from EtOH. M.p. 61°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Volatile in steam.

*Acetyl*: needles. M.p. 74°. Sol. boiling H<sub>2</sub>O, most org. solvents.

*p*-Toluenesulphonyl: prisms from EtOH. M.p. 123–4°. Sol. AcOH. Spar. sol. MeOH, EtOH.

Ullmann, Fitzenkam, *Ber.*, 1905, **38**, 3790.

Spiegel, Munblit, Kaufmann, *Ber.*, 1906, **39**, 3241.

### 5-Nitro-*o*-cresol.

Needles + H<sub>2</sub>O from H<sub>2</sub>O or EtOH.Aq., yellow plates from C<sub>6</sub>H<sub>6</sub>. M.p. 30–40°, anhyd. 96°. B.p. 186–90°/9 mm.

*Me ether*: needles from EtOH or pet. ether. M.p. 64°. Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, CCl<sub>4</sub>.

*Et ether*: needles from EtOH.Aq. M.p. 71°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, hot ligroin.

*Benzoyl*: m.p. 128°. Mod. sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, CCl<sub>4</sub>. Spar. sol. EtOH, Et<sub>2</sub>O, pet. ether.

*p*-Toluenesulphonyl: cryst. from EtOH. M.p. 107°.

Gibson, *J. Chem. Soc.*, 1925, **127**, 44.

### 6-Nitro-*o*-cresol.

Yellowish needles from H<sub>2</sub>O. M.p. 147°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O. Sweet taste.

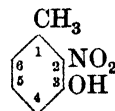
*Me ether*: needles from MeOH.Aq. M.p. 52–3°.

*p*-Toluenesulphonyl: plates from EtOH. M.p. 94°.

Noelting, *Ber.*, 1904, **37**, 1020.

Simonsen, Nayak, *J. Chem. Soc.*, 1915, **107**, 832.

### 2-Nitro-*m*-cresol



C<sub>7</sub>H<sub>7</sub>O<sub>3</sub>N

MW, 153

Yellow needles from pet. ether. M.p. 41°. Sol. usual org. solvents. Odour of iodoform.

*Me ether*: C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 167. Yellow plates from EtOH. M.p. 54°. Volatile in steam.

*Acetyl*: needles from EtOH. M.p. 59°.

*Benzoyl*: needles from EtOH. M.p. 79°.

Sol.  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{AcOEt}$ ,  $\text{CCl}_4$ . Spar. sol.  $\text{EtOH}$ , pet. ether.

Barger, Schlittler, *Helv. Chim. Acta*, 1932, **15**, 389.

Gibson, *J. Chem. Soc.*, 1923, **123**, 1269.

Cf. also Corbellini, Ravazzoni, *Atti accad. Lincei*, 1931, **13**, 132.

#### 4-Nitro-*m*-cresol.

Yellow plates from  $\text{Et}_2\text{O}$  or  $\text{C}_6\text{H}_6$ . M.p.  $56^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ . Volatile in steam.

*Me ether*: cryst. M.p.  $62^\circ$ .

*Et ether*:  $\text{C}_9\text{H}_{11}\text{O}_3\text{N}$ . MW, 181. Prisms from pet. ether. M.p.  $55^\circ$  ( $50-1^\circ$ ).

*Acetyl*: plates from  $\text{EtOH}$ . M.p.  $48^\circ$ .

*Benzoyl*: prisms. M.p.  $77^\circ$ .

Gibson, *J. Chem. Soc.*, 1923, **123**, 1269.

#### 5-Nitro-*m*-cresol.

Pale yellow cryst. +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ , cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $60-2^\circ$ , anhyd.  $90-1^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Less sol.  $\text{C}_6\text{H}_6$ . Non-volatile in steam.

*Me ether*: cryst. M.p.  $70^\circ$ . *Et ether*: cryst. from  $\text{Et}_2\text{O}$ . M.p.  $53-4^\circ$ .

Neville, Winther, *Ber.*, 1882, **15**, 2986.

Blanksma, *Rec. trav. chim.*, 1908, **27**, 25.

Hoshino, Kotake, *Ann.*, 1935, **516**, 76.

#### 6-Nitro-*m*-cresol.

Needles from  $\text{H}_2\text{O}$ . M.p.  $129^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{H}_2\text{O}$ .

*Me ether*: needles from ligroin. M.p.  $55^\circ$ . Sol. most org. solvents.

*Et ether*: needles from  $\text{EtOH}$ . M.p.  $45^\circ$ .

*Acetyl*: fine needles from  $\text{EtOH}$ . M.p.  $34^\circ$ .

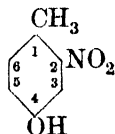
*Benzoyl*: needles. M.p.  $74^\circ$ .

Staedel, *Ann.*, 1883, **217**, 51.

Gibson, *J. Chem. Soc.*, 1923, **123**, 1269.

See also last reference above.

#### 2-Nitro-*p*-cresol



$\text{C}_7\text{H}_7\text{O}_3\text{N}$

MW, 153

Yellow prisms from  $\text{Et}_2\text{O}$ . M.p.  $79^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ , ligroin.

*Me ether*:  $\text{C}_8\text{H}_9\text{O}_3\text{N}$ . MW, 167. Yellow prisms. M.p.  $17^\circ$ . B.p.  $266-7^\circ$ . Volatile in steam.

Holleman, Hoeflake, *Rec. trav. chim.*, 1916, **36**, 272.

Copisarow, *J. Chem. Soc.*, 1929, 251.

#### 3-Nitro-*p*-cresol.

Yellow needles from  $\text{EtOH.Aq.}$  M.p.  $36.5^\circ$  ( $32^\circ$ ). B.p.  $125^\circ/22\text{ mm.}$ ,  $114.5^\circ/7.5\text{ mm.}$  Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , and most other org. solvents. Spar. sol.  $\text{H}_2\text{O}$ .

*Me ether*: pale yellow cryst. M.p.  $8.5^\circ$ . B.p.  $274^\circ$ ,  $159^\circ/15\text{ mm.}$   $D_4^{25}$  1.2025.  $n_D^{25}$  1.5536. Sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{EtOH}$ .

*Et ether*:  $\text{C}_9\text{H}_{11}\text{O}_3\text{N}$ . MW, 181. B.p.  $275-85^\circ$  decomp.

*Benzoyl*: prisms. M.p.  $100-1^\circ$ .

Baranger, *Bull. soc. chim.*, 1931, **49**, 1214.

Derick, Ralph, Flett, U.S.P., 1,394,150, (*Chem. Abstracts*, 1922, **16**, 423).

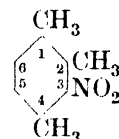
Brasch, Freyss, *Ber.*, 1891, **24**, 1960.

Neunhöfer, Kölbel, *Ber.*, 1935, **68**, 255.

#### Nitrocresotic Acid.

See Nitrohydroxytoluic Acid.

#### 3-Nitro- $\psi$ -cumene



$\text{C}_9\text{H}_{11}\text{O}_2\text{N}$

MW, 165

Cryst. M.p.  $30^\circ$ .

Huender, *Rec. trav. chim.*, 1915, **34**, 10.

Mayer, *Ber.*, 1887, **20**, 971.

#### 5-Nitro- $\psi$ -cumene.

Yellowish needles. M.p.  $71^\circ$  ( $70^\circ$ ). B.p.  $265^\circ$ .

Schultz, *Ber.*, 1909, **42**, 3605.

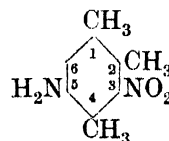
Huender, *Rec. trav. chim.*, 1915, **34**, 14.

#### 6-Nitro- $\psi$ -cumene.

Prisms from  $\text{EtOH}$ . M.p.  $20^\circ$ .

Edler, *Ber.*, 1885, **18**, 629.

#### 3-Nitro- $\psi$ -cumidine



$\text{C}_9\text{H}_{12}\text{O}_2\text{N}_2$

MW, 180

Golden-yellow needles. M.p.  $138^\circ$ . Sol.  $\text{EtOH}$ . Spar. sol. boiling  $\text{H}_2\text{O}$ . Sublimes. Volatile in steam.

Huender, *Rec. trav. chim.*, 1915, **34**, 17.

Blanksma, *Rec. trav. chim.*, 1905, **24**, 48.

#### 6-Nitro- $\psi$ -cumidine.

Light red needles from  $\text{EtOH.Aq.}$  M.p.  $46-7^\circ$ . Very sol.  $\text{Et}_2\text{O}$ .

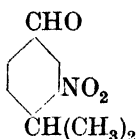
*Acetyl*: needles or prisms from EtOH. M.p. 202–4° (199°). Mod. sol. EtOH, AcOH, CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O.

*Propionyl*: cryst. from EtOH. M.p. 167°. Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, Py, isoamyl alcohol, CCl<sub>4</sub>. Spar. sol. Et<sub>2</sub>O, ligroin.

Edler, *Ber.*, 1885, 18, 629.

Bogert, Bender, *J. Am. Chem. Soc.*, 1914, 36, 573.

### 3-Nitrocuminaldehyde



C<sub>10</sub>H<sub>11</sub>O<sub>3</sub>N

MW, 193

Yellow cryst. from EtOH. M.p. 54°.

*Oxime*: needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 74–6°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O, ligroin.

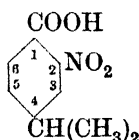
*Semicarbazone*: needles from EtOH. M.p. 222°. Sol. boiling EtOH. Spar. sol. AcOH, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O, Et<sub>2</sub>O, ligroin. Turns yellow in air.

*Phenylhydrazone*: m.p. 123°.

Lippmann, Strecker, *Ber.*, 1879, 12, 76.

Pizzuti, *Gazz. chim. ital.*, 1910, 40, ii, 236.

### 2-Nitrocuminic Acid



C<sub>10</sub>H<sub>11</sub>O<sub>4</sub>N

MW, 209

Plates from 50% AcOH or ligroin. M.p. 99°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin.

Widman, *Ber.*, 1886, 19, 269.

### 3-Nitrocuminic Acid.

Prisms from EtOH. M.p. 158–9°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.  $k = 2.15 \times 10^{-4}$  at 25°.

*Me ester*: C<sub>11</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 223. Cryst. from EtOH. M.p. 64°. Sol. warm EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Et ester*: C<sub>12</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 237. Liq. B.p. 290° decomp.

*Nitrile*: C<sub>10</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>. MW, 190. Cryst. from EtOH. M.p. 71°. Very sol. EtOH, Et<sub>2</sub>O.

Widman, *Ber.*, 1888, 21, 2232.

Alexejew, *J. Russ. Phys.-Chem. Soc.*, 1885, 17, 112.

### Nitrocycloacetic Acid



C<sub>3</sub>H<sub>2</sub>O<sub>4</sub>N<sub>2</sub>

MW, 130

*Me ester*: C<sub>4</sub>H<sub>4</sub>O<sub>4</sub>N<sub>2</sub>. MW, 144. Needles + 1H<sub>2</sub>O. M.p. 76°. Sol. H<sub>2</sub>O, MeOH. Spar. sol. Et<sub>2</sub>O. *K salt*: leaflets from H<sub>2</sub>O. M.p. 264–6° decomp. *Hydrazine salt*: N<sub>2</sub>H<sub>4</sub>·C<sub>4</sub>H<sub>4</sub>O<sub>4</sub>N<sub>2</sub>. Cryst. from MeOH. M.p. 168°. Very sol. H<sub>2</sub>O.

*Et ester*: C<sub>5</sub>H<sub>6</sub>O<sub>4</sub>N<sub>2</sub>. MW, 158. Not obtained pure. Prisms from H<sub>2</sub>O. Sol. EtOH. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. *K salt*: cryst. M.p. 240°.

*Amide*: fulminuric acid. C<sub>3</sub>H<sub>3</sub>O<sub>3</sub>N<sub>3</sub>. MW, 129. Prisms from EtOH. M.p. 145° (136–49° decomp.). Sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin. *NH<sub>4</sub> salt*: prisms. Spar. sol. cold H<sub>2</sub>O. Insol. EtOH, Et<sub>2</sub>O. *K salt*: prisms. Sol. 10 parts cold H<sub>2</sub>O. Insol. EtOH, Et<sub>2</sub>O.

*Hydrazide*: colourless needles + 1H<sub>2</sub>O or yellow anhyd. prisms. M.p. above 285°.

Ulpiani, *Gazz. chim. ital.*, 1912, 42, i, 225.

Darapsky, Hillers, *J. prakt. Chem.*, 1915, 92, 324.

Conrad, Schulze, *Ber.*, 1909, 42, 740.

Nef, *Ann.*, 1894, 280, 329.

### Nitrocyanobenzoic Acid.

*See under* Nitrophthalic Acid.

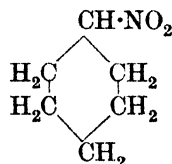
### Nitro-α-cyanocinnamic Acid.

*See under* Nitrobenzylidenemalononic Acid.

### Nitrocyanophenol.

*See under* Nitrohydroxybenzoic Acid and Nitrosalicylic Acid.

### Nitrocyclohexane



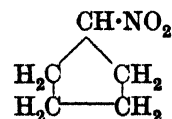
C<sub>6</sub>H<sub>11</sub>O<sub>2</sub>N

MW, 129

F.p. –34°. B.p. 205.5–206°, 109°/40 mm. D<sub>4</sub><sup>0</sup> 1.0853, D<sub>4</sub><sup>19</sup> 1.0680.  $n_D^{19}$  1.4612. SnCl<sub>2</sub> + HCl → cyclohexanone oxime. Sn + HCl → cyclohexanone + aminocyclohexane.

Nametkin, *Ber.*, 1909, 42, 1372.

### Nitrocyclopentane

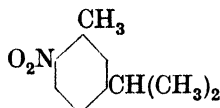


C<sub>5</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 115

B.p. 90–1°/40 mm.  $D_4^{23}$  1.0776.  $n_D^{23}$  1.4518.  
 $\text{HNO}_3 \rightarrow$  glutaric acid.

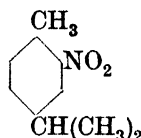
Nametkin, *Chem. Zentr.*, 1912, I, 1702.

6-Nitro-*m*-cymene

$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

Liq. B.p. 230–40°.

Meyer, Bernhauer, *Monatsh.*, 1929, 53  
 and 54, 741.

2-Nitro-*p*-cymene

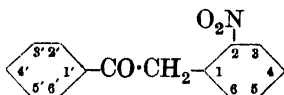
$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

Liq. B.p. 130–5°/14 mm., 129–32°/15 mm.  
 $D_4^{20}$  1.0744.  $n_D^{20}$  1.53093.

Inoue, Horiguchi, *Chem. Abstracts*, 1933,  
 27, 3464.

Demonbreun, Kremers, *Chem. Abstracts*,  
 1923, 17, 3906.

**2-Nitrodeoxybenzoin** ( $\omega$ -o-Nitrophenylaceto-  
 phenone, phenyl o-nitrobenzyl ketone)



$\text{C}_{14}\text{H}_{11}\text{O}_3\text{N}$  MW, 241

Needles from  $\text{C}_6\text{H}_6$ -ligroin. M.p. 73–4°. Sol.  
 $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{AcOH}$ .  $\text{CrO}_3 \rightarrow$  2-nitro-  
 benzil.  $\text{Zn dust} + \text{NH}_3\text{.Aq.} \rightarrow$  2-phenylindole.  
 $\text{Alc. KOH} \rightarrow$  blue col.

*Oxime*: needles from  $\text{EtOH}$ . M.p. 118°.  
 $\text{Alkalis} \rightarrow$  red sols.

List, *Ber.*, 1893, 26, 2453.

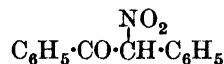
**4-Nitrodeoxybenzoin** ( $\omega$ -p-Nitrophenylaceto-  
 phenone, phenyl p-nitrobenzyl ketone).

Prisms from  $\text{EtOH}$ . M.p. 145° (140–2°).  
 Sol. 597 parts cold, 22.5 parts boiling 95%  $\text{EtOH}$ .  
 Mod. sol. boiling  $\text{AcOH}$ . Spar. sol. boiling  $\text{Et}_2\text{O}$ .  
 $\text{CrO}_3 \rightarrow$  4-nitrobenzil.  $\text{Alc. KOH} \rightarrow$  violet  
 col.

*Oxime*: needles from  $\text{EtOH}$ . M.p. 107° (105°).

See previous reference and also  
 Golubeff, *Ber.*, 1878, 11, 1939.

**$\alpha$ -Nitrodeoxybenzoin** ( $\omega$ -Nitro- $\omega$ -phenyl-  
 acetophenone)



$\text{C}_{14}\text{H}_{11}\text{O}_3\text{N}$  MW, 241

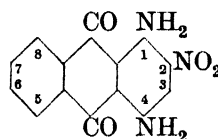
*Di-Me acetal*: needles from  $\text{MeOH}$ . M.p.  
 202–3°.

Wieland, Blümich, *Ann.*, 1921, 424, 106.

**Nitrodiaminoanisole.**

See under Nitrodiaminophenol.

**2-Nitro-1 : 4-diaminoanthraquinone**



$\text{C}_{14}\text{H}_9\text{O}_4\text{N}_3$  MW, 283

Greenish-blue needles. Sol. inert solvents with  
 blue col. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  colourless sol.  $\rightarrow$   
 blue on warming with boric acid.

$\text{N} : \text{N}'$ -*Diacetyl*: yellowish-brown needles from  
 $\text{AcOH}$ . M.p. 237° decomp. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$   
 pale yellow sol.  $\rightarrow$  blue on adding formalde-  
 hyde.

$\text{N} : \text{N}'$ -*Dibenzoyl*: orange needles. Sol. conc.  
 $\text{H}_2\text{SO}_4$  with red col.

M.L.B., D.R.P., 254,185, (*Chem. Zentr.*,  
 1913, I, 197).

Bayer, D.R.P., 267,445, (*Chem. Zentr.*,  
 1914, I, 88).

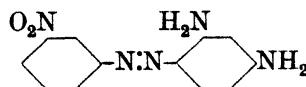
**5-Nitro-1 : 4-diaminoanthraquinone.**

$\text{N} : \text{N}'$ -*Diacetyl*: red cryst. Py sol. is yel-  
 lowish-red. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*Sulphate*: dark violet prisms from  $\text{PhNO}_2$ .  
 Sol. Py with violet-blue col.

Bayer, D.R.P., 268,984, (*Chem. Zentr.*,  
 1914, I, 588).

**3'-Nitro-2 : 4-diaminoazobenzene**



$\text{C}_{12}\text{H}_{11}\text{O}_2\text{N}_5$  MW, 257

Yellow cryst. from  $\text{EtOH}$ .Aq. M.p. 204°.

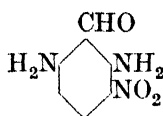
*Tetra-Me*:  $\text{C}_{16}\text{H}_{19}\text{O}_2\text{N}_5$ . MW, 313. Free  
 base cannot be cryst. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .  
 Insol.  $\text{H}_2\text{O}$ .  $\text{B.HCl}$ : dark violet needles +  
 $\text{H}_2\text{O}$  from dil.  $\text{HCl}$ . M.p. anhyd. 198°. Sol.  
 $\text{H}_2\text{O}$  with yellow col.

Leonhardt, D.R.P., 37,021.

Sachs, Appenzeller, *Ber.*, 1908, 41, 110.

### 3-Nitro-2 : 6-diaminobenzaldehyde

#### 3-Nitro-2 : 6-diaminobenzaldehyde



$C_7H_7O_3N_3$

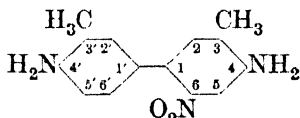
MW, 181

Yellow needles from trichlorobenzene. M.p. 250-1°.

I.G., E.P., 339,699, (*Chem. Zentr.*, 1931, II, 1925); D.R.P., 521,724, (*Chem. Zentr.*, 1931, I, 3722).

Kalischer, Ritter, Honold, U.S.P., 1,876,955, (*Chem. Abstracts*, 1933, 27, 993).

**6-Nitro-4 : 4'-diamino-3 : 3'-dimethyldiphenyl** (6-Nitro-4 : 4'-diamino-3 : 3'-ditolyl, 6-nitro-3 : 3'-dimethylbenzidine, 6-nitro-o-tolidine)



$C_{14}H_{15}O_2N_3$

MW, 257

Red needles from toluene. M.p. 156°. Sol. AcOH. Mod. sol.  $C_6H_6$ . Less sol. EtOH. Spar. sol. Et<sub>2</sub>O. Insol. ligroin.

4 : 4'-N-Diacetyl : yellow needles. M.p. 290°.

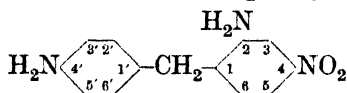
4 : 4'-N-Dibenzylidene : leaflets. M.p. 147°.

Löwenherz, *Ber.*, 1892, 25, 1032.

**Nitro-4 : 4'-diaminodiphenyl.**

See Nitrobenzidine.

**4-Nitro-2 : 4'-diaminodiphenylmethane**



$C_{13}H_{13}O_2N_3$

MW, 243

Orange needles from boiling EtOH. M.p. 157-8°. Sol. about 23 parts boiling EtOH.

B<sub>2</sub>HCl : leaflets from H<sub>2</sub>O. M.p. 262° decomp.

*Diacetyl deriv.* : needles from boiling MeOH. M.p. 239-40°. 1-2 parts sol. 100 parts boiling MeOH.

*Tetra-acetyl* : prisms from boiling EtOH. M.p. 201.5-202.5°. Sol. 100 parts boiling EtOH.

King, *J. Chem. Soc.*, 1920, 117, 989.

**2-Nitro-4 : 4'-diaminodiphenylmethane.**

Yellow needles from EtOH. M.p. 100-1°.

4 : 4'-N-Tetra-Me :  $C_{17}H_{21}O_2N_3$ . MW, 299. Deep red needles from EtOH. M.p. 96-96.5° (95°). Sol. warm EtOH,  $C_6H_6$ , AcOH. Spar. sol. ligroin.

### 126 2''-Nitro-4 : 4'-diaminotriphenylmethane

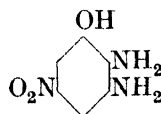
4 : 4'-N-Tetra-Et :  $C_{21}H_{29}O_2N_3$ . MW, 355. Red prisms. M.p. 79-80°.

Epstein, D.R.P., 139,989, (*Chem. Zentr.*, 1908, I, 798).

Bayer, D.R.P., 79,250.

Ullmann, Marié, *Ber.*, 1901, 34, 4314.

**5-Nitro-2 : 3-diaminophenol** (5-Nitro-3-hydroxy-o-phenylenediamine)



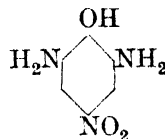
$C_6H_7O_3N_3$

MW, 169

*Me ether* : 5-nitro-2 : 3-diaminoanisole.  $C_7H_9O_3N_3$ . MW, 183. Dark red needles from H<sub>2</sub>O. M.p. 131-2°.

Borsche, *Ber.*, 1917, 50, 1348.

**4-Nitro-2 : 6-diaminophenol** (5-Nitro-2-hydroxy-m-phenylenediamine)



$C_6H_7O_3N_3$

MW, 169

Yellow needles or plates + H<sub>2</sub>O. Mod. sol. EtOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O.

N-Acetyl : cryst. Sol. AcOH. Mod. sol. Et<sub>2</sub>O. Spar. sol. EtOH.

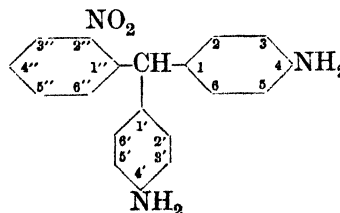
Griess, *Ann.*, 1870, 154, 202.

Cassella, D.R.P., 161,341, (*Chem. Zentr.*, 1905, II, 181).

**Nitrodiaminotoluene.**

See Nitrotolylene-diamine.

**2''-Nitro-4 : 4'-diaminotriphenylmethane**



$C_{19}H_{17}O_2N_3$

MW, 319

Yellowish-red cryst. from  $C_6H_6$ -ligroin.

4 : 4'-N-Di-Me :  $C_{21}H_{21}O_2N_3$ . MW, 347. Golden-yellow prisms from EtOH- $C_6H_6$ . M.p. 159-60°. Mod. sol. H<sub>2</sub>O, ligroin. Spar. sol. EtOH.

4 : 4'-N-Di-Et :  $C_{23}H_{25}O_2N_3$ . MW, 375. Yel.

**3''-Nitro-4:4'-diaminotriphenylmethane 127**

lowish-red cryst. from  $C_6H_6$ -EtOH. M.p. 109-10°.

Fischer, Schmidt, *Ber.*, 1884, 17, 1889.

Renouf, *Ber.*, 1883, 16, 1304.

**3''-Nitro-4:4'-diaminotriphenylmethane.**

Yellow cryst. from  $Et_2O$ -ligroin. M.p. 136°. Sol. EtOH,  $Et_2O$ . Less sol.  $C_6H_6$ . Spar. sol. ligroin. Insol.  $H_2O$ .

4:4'-N-*Di-Me*: yellow prisms from EtOH, golden needles from  $C_6H_6$ . M.p. 152°. Sol.  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ , ligroin. *Dimethiodide*: needles from EtOH. M.p. 225° decomp.

4:4'-N-*Di-Et*: yellow needles with green fluor. from EtOH. M.p. 95-6°.

Fischer, Ziegler, *Ber.*, 1880, 13, 671.

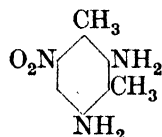
**4''-Nitro-4:4'-diaminotriphenylmethane.**

Yellow cryst. from EtOH. Sol.  $Me_2CO$ ,  $C_6H_6$ ,  $CHCl_3$ . Less sol. EtOH,  $Et_2O$ . Spar. sol. ligroin. Insol.  $H_2O$ .

4:4'-N-*Di-Me*: golden-yellow plates from toluene-EtOH. M.p. 176-7°. Spar. sol. EtOH, ligroin. Insol.  $H_2O$ . *Dimethiodide*: yellow needles from  $H_2O$ . M.p. 220° decomp. Spar. sol. EtOH.

4:4'-N-*Di-Et*: reddish-brown plates from EtOH.Aq. M.p. 113°.

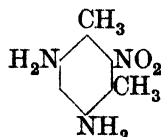
Fischer, *Ber.*, 1881, 14, 2526; 1882, 15, 677.

**6-Nitro-2:4-diamino-m-xylene (5-Nitro-2:4-dimethyl-m-phenylenediamine)**

$C_8H_{11}O_2N_3$  MW, 181

Orange-yellow needles from  $H_2O$ . M.p. 151-2°. Sol. EtOH. Spar. sol.  $H_2O$ .

Noelting, Thesmar, *Ber.*, 1902, 35, 630.

**2-Nitro-4:6-diamino-m-xylene (5-Nitro-4:6-dimethyl-m-phenylenediamine)**

$C_8H_{11}O_2N_3$  MW, 181

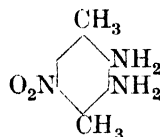
Red prisms from EtOH or  $H_2O$ . M.p. 212-13°. Sol. EtOH. Mod. sol. boiling  $H_2O$ . Sublimes.

**4-Nitrodiazoaminobenzene**

N:N'-*Di-Et*:  $C_{12}H_{10}O_2N_3$ . MW, 237. Yellow cryst. Sol. EtOH,  $Et_2O$ .

Fittig, Velguth, *Ann.*, 1868, 148, 6.

Bussenius, Eisenstuck, *Ann.*, 1860, 113, 159.

**5-Nitro-2:3-diamino-p-xylene (4-Nitro-3:6-dimethyl-o-phenylenediamine)**

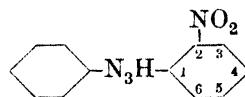
$C_8H_{11}O_2N_3$  MW, 181

Light red needles from  $C_6H_6$  or EtOH.Aq. M.p. 169°. Sol.  $Et_2O$ , AcOH. Mod. sol. hot  $H_2O$ . Spar. sol. pet. ether.

2-N-*Phenyl*: 4-nitro-6-amino-2:5-dimethyldiphenylamine.  $C_{14}H_{12}O_2N_3$  MW, 257. Orange-yellow leaflets. M.p. 171°. Sol. EtOH, AcOH. Less sol.  $C_6H_6$ . Spar. sol. pet. ether. N'-*Acetyl*: yellow leaflets from  $C_6H_6$ -pet. ether. M.p. 203°.

Fries, Noll, *Ann.*, 1912, 389, 374.

Fries, Arnemann, *Ann.*, 1927, 454, 160.

**2-Nitrodiazoaminobenzene**

$C_{12}H_{10}O_2N_4$  MW, 242

Orange-yellow needles from EtOH. M.p. 104.5-105°. Sol.  $CHCl_3$ ,  $Me_2CO$ , AcOH,  $C_6H_6$ .

Bamberger, *Ber.*, 1895, 28, 237.

**3-Nitrodiazoaminobenzene.**

Yellow prisms from  $Et_2O$ . M.p. 131°.

Goldschmidt, Molinari, *Ber.*, 1888, 21, 2572.

**4-Nitrodiazoaminobenzene.**

Yellow needles from  $C_6H_6$ . M.p. 148° decomp. N-*Me*:  $C_{13}H_{12}O_2N_4$ . MW, 256. Yellow cryst. from ligroin. M.p. 134°. Spar. sol. hot EtOH. N-*Et*:  $C_{14}H_{14}O_2N_4$ . MW, 270. Yellow cryst. from ligroin. M.p. 115-16°. Sol.  $Et_2O$ ,  $C_6H_6$ .  $CHCl_3$ , hot EtOH.

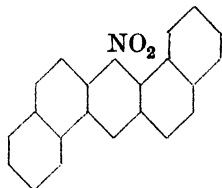
N-*Phenyl*:  $C_{18}H_{14}O_2N_4$ . MW, 318. Brownish-red. M.p. 63°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

Nölting, Binder, *Ber.*, 1887, 20, 3014.

Bamberger, *Ber.*, 1895, 28, 839.



**9-Nitro-1 : 2 : 5 : 6-dibenzanthracene** (9-Nitro-1 : 2 : 5 : 6-dinaphthanthracene)

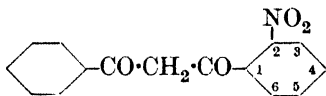
C<sub>22</sub>H<sub>18</sub>O<sub>2</sub>N

MW, 323

Orange-yellow needles from xylene. M.p. 217–18°.

Cook, *J. Chem. Soc.*, 1931, 3276.

**2-Nitrodibenzoylmethane**

C<sub>15</sub>H<sub>11</sub>O<sub>4</sub>N

MW, 269

Needles from EtOH. M.p. 116°. Alc. FeCl<sub>3</sub> → red col.

Gabriel, Gerhard, *Ber.*, 1921, 54, 1615.

**3-Nitrodibenzoylmethane.**

Yellowish cryst. from AcOH. M.p. 135°. Sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOH. Less sol. MeOH, EtOH. Spar. sol. Et<sub>2</sub>O, ligroin. Alc. FeCl<sub>3</sub> → brownish-violet col.

*Mono-semicarbazone*: cryst. from MeOH.Aq. M.p. 120–5° decomp.

Bodforss, *Ber.*, 1916, 49, 2804; 1918, 51, 215.

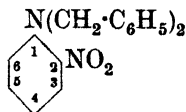
**4-Nitrodibenzoylmethane.**

Yellow needles from EtOH. M.p. 160°. Sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. CHCl<sub>3</sub>, EtOH. Practically insol. Et<sub>2</sub>O. FeCl<sub>3</sub> → red col.

*Di-Me acetal*: C<sub>17</sub>H<sub>17</sub>O<sub>5</sub>N. MW, 315. Plates from EtOH or pet. ether. M.p. 91°. Mod. sol. usual solvents.

Wieland, *Ber.*, 1904, 37, 1151.

***o*-Nitro-*N*-dibenzylaniline**

C<sub>20</sub>H<sub>18</sub>O<sub>2</sub>N<sub>2</sub>

MW, 318

Yellow plates from pet. ether. M.p. 32–3°. Sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, AcOH, pet. ether.

Desai, *J. Indian Chem. Soc.*, 1928, 5, 428.

***m*-Nitro-*N*-dibenzylaniline.**

Golden-yellow needles from EtOH. M.p. 73–4°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, toluene. *B,HCl*: needles from AcOH. M.p. 140–2°.

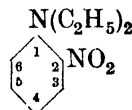
See previous reference.

***p*-Nitro-*N*-dibenzylaniline.**

Yellow plates from AcOH. M.p. 132–3°. Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Mod. sol. AcOH. Almost insol. cold EtOH.

See previous reference.

***o*-Nitro-*N*-diethylaniline**

C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub>

MW, 194

Orange-yellow oil with pungent odour. Distils in vacuo. Sol. EtOH, Et<sub>2</sub>O. Less sol. H<sub>2</sub>O.

*B,HCl*: cryst. from EtOH. Decomp. at 156°.

*B,HBr*: plates from EtOH. Decomp. at 160°.

Hygroscopic.

*B,HI*: needles from EtOH. M.p. 112°.

Hygroscopic.

*B,H<sub>2</sub>SO<sub>4</sub>*: plates from EtOH. M.p. 143°.

*Picrate*: golden-yellow plates. M.p. 119–20°.

Sol. hot EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

Weissenberger, *Monatsh.*, 1912, 33, 830.

***m*-Nitro-*N*-diethylaniline.**

Dark yellow oil. B.p. 288–90°.

*Picrate*: cryst. from EtOH. M.p. 138°.

Noelting, Stricker, *Ber.*, 1886, 19, 550.

***p*-Nitro-*N*-diethylaniline.**

Yellow needles from ligroin, plates from EtOH. M.p. 77–8°. Sol. hot EtOH. Spar. sol. ligroin.

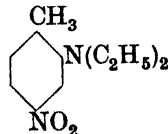
Hollemann, de Mooy, *Rec. trav. chim.*, 1916, 35, 32.

Davies, *Bull. soc. chim.*, 1935, 2, 295.

**2-Nitrodiethyl Ether.**

See under 2-Nitroethyl Alcohol.

**4-Nitro-*N*-diethyl-*o*-toluidine**

C<sub>11</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>

MW, 208

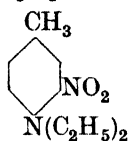
Yellow oil. B.p. 283° (295–7°/727 mm.). Sol. EtOH, Et<sub>2</sub>O. Volatile in steam.

Ullmann, Mühlhauser, *Ber.*, 1902, 35, 335.

Möhlau, Klimmer, Kahl, *Chem. Zentr.*, 1902, II, 378.

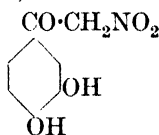
**3-Nitro-N-diethyl-p-toluidine**

129

**4'-Nitro-3 : 4-dihydroxyazobenzene****3-Nitro-N-diethyl-p-toluidine** $C_{11}H_{16}O_2N_2$ 

MW, 208

Yellow oil. B.p. 101-2°/0.7 mm.

Schmidt, Fischer, *Ber.*, 1920, **53**, 1533.**ω-Nitro-3 : 4-dihydroxyacetophenone (4-Nitroacetylcatechol)** $C_8H_7O_5N$ 

MW, 197

Yellowish cryst. M.p. 188° decomp. Sol. 10 parts hot  $H_2O$ . Sol. EtOH,  $Me_2CO$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Dil. NaOH  $\rightarrow$  intense red sol.  $FeCl_3 \rightarrow$  green col.

*Di-Me ether*:  $C_{10}H_{11}O_5N$ . MW, 225. Needles from AcOEt. M.p. 144°. Sol. AcOEt. Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

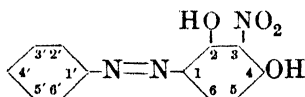
M.L.B., D.R.P., 195,814, (*Chem. Zentr.*, 1908, I, 1225).

**Nitrodihydroxyaniline.**

See Nitroaminocatechol, Nitroaminoresorcinol and Nitroaminohydroquinone.

**Nitrodihydroxyanthraquinone.**

See Nitroalizarin, Nitrochrysazin, Nitrohystazarin, and Nitroquinizarin.

**3-Nitro-2 : 4-dihydroxyazobenzene** $C_{12}H_9O_4N_3$ 

MW, 259

Dark red needles from AcOH. M.p. 171°. Sol. alkalis with orange-red col.

Kauffmann, de Pay, *Ber.*, 1906, **39**, 327.

**2'-Nitro-2 : 4-dihydroxyazobenzene (o-Nitrobenzeneazoresorcinol).**

Red needles from EtOH. M.p. 180°. Forms dark red Ba salt.

Elbs *et al.*, *J. prakt. Chem.*, 1924, **108**, 217.

**4'-Nitro-2 : 4-dihydroxyazobenzene (p-Nitrobenzeneazoresorcinol).**

Red cryst. powder from MeOH. M.p. 199-200°. Spar. sol. AcOH, toluene, boiling EtOH.

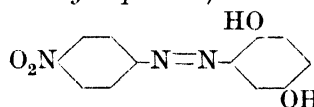
Dict. of Org. Comp.—III.

Forms brownish-red Na salt insol.  $H_2O$ . Reagent for magnesium.

Meldola, *J. Chem. Soc.*, 1885, **47**, 660.

Orton, Everatt, *J. Chem. Soc.*, 1908, **93**, 1018.

Fischer, Taurinsch, *Ber.*, 1931, **64**, 236.

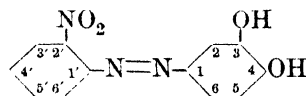
**4'-Nitro-2 : 5-dihydroxyazobenzene (p-Nitrobenzeneazohydroquinone)** $C_{12}H_9O_4N_3$ 

MW, 259

Plates from EtOH. Decomp. at 185-90°.

*5-Benzoyl*: brown needles from AcOH.Aq. M.p. 195-7°.

Witt, Johnson, *Ber.*, 1893, **26**, 1910.

**2'-Nitro-3 : 4-dihydroxyazobenzene (o-Nitrobenzeneazocatechol)** $C_{12}H_9O_4N_3$ 

MW, 259

*3-Me ether*:  $C_{13}H_{11}O_4N_3$ . MW, 273. Red cryst. M.p. 144°. Readily sol. org. solvents. Sol. alkalis with wine-red col.

*Di-Me ether*: o-nitrobenzeneazoveratrol.  $C_{14}H_{13}O_4N_3$ . MW, 287. Red cryst. M.p. 152°. Sol. most org. solvents.

Colombano, Leonardi, *Gazz. chim. ital.*, 1907, **37**, ii, 467.

**3'-Nitro-3 : 4-dihydroxyazobenzene (m-Nitrobenzeneazocatechol).**

*3-Me ether*: red needles from EtOH.Aq. M.p. 124°. *Acetyl*: cryst. from ligroin. M.p. 95-7°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

See previous reference.

**4'-Nitro-3 : 4-dihydroxyazobenzene (p-Nitrobenzeneazocatechol).**

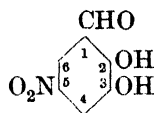
Red cryst. Sol. in alkalis with blue col., in ammonia and alkali carbonates with violet col., and in conc.  $H_2SO_4$  with red col.

*3-Me ether*: brown cryst. from EtOH.Aq. M.p. 125-35°.

*Diacetyl*: orange-red needles from MeOH. M.p. 126-7°.

Witt, Mayer, *Ber.*, 1893, **26**, 1074.

## 5-Nitro-2 : 3-dihydroxybenzaldehyde

 $C_7H_5O_5N$ 

MW, 183

3-Me ether:  $C_8H_7O_5N$ . MW, 197. Pale yellow needles from EtOH. M.p.  $142^\circ$ .

Di-Me ether: see 5-Nitro-2 : 3-dimethoxybenzaldehyde.

3-Me-2-Et ether:  $C_{10}H_{11}O_5N$ . MW, 225. Pale yellow plates. M.p.  $137^\circ$ . p-Me-anil: pale yellow needles from EtOH. M.p.  $148^\circ$ .

2-Me-3-Et ether: needles from  $CHCl_3$ . M.p.  $118-5^\circ$ .

3-Et ether:  $C_9H_9O_5N$ . MW, 211. Pale yellow cryst. from EtOH. M.p.  $158^\circ$ .

Di-Et ether:  $C_{11}H_{13}O_5N$ . MW, 239. Pale yellow needles. M.p.  $71^\circ$ . p-Me-anil: needles from EtOH. M.p.  $105-6^\circ$ .

Davies, *J. Chem. Soc.*, 1923, **123**, 1580.

Davies, Rubenstein, *ibid.*, 2844.

Rubenstein, *J. Chem. Soc.*, 1925, 2268.

## 6-Nitro-2 : 3-dihydroxybenzaldehyde.

3-Me ether: 2-benzenesulphonyl, yellow needles from EtOH. M.p.  $145^\circ$ . Turns greenish-blue in sunlight.

Di-Me ether: see 6-Nitro-2 : 3-dimethoxybenzaldehyde.

3-Me-2-Et ether: prisms from EtOH.Aq. M.p.  $57^\circ$ . p-Nitrophenylhydrazone: brown needles. M.p.  $188-9^\circ$ . p-Me-anil: golden prisms. M.p.  $88^\circ$ .

Di-Et ether: needles from EtOH.Aq. M.p.  $75-6^\circ$ . Turns green in air. p-Nitrophenylhydrazone: bright yellow needles. M.p.  $268-70^\circ$ .

See previous references.

## Nitro-2 : 4-dihydroxybenzaldehyde.

See Nitroresorcylic Aldehyde.

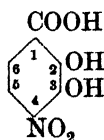
## Nitro-2 : 5-dihydroxybenzaldehyde.

See Nitrogentic Acid.

## Nitro-3 : 4-dihydroxybenzaldehyde.

See Nitroprotocatechuic Aldehyde.

## 4-Nitro-2 : 3-dihydroxybenzoic Acid

 $C_7H_5O_6N$ 

MW, 199

2-Me ether:  $C_8H_7O_6N$ . MW, 213. Leaflets from  $H_2O$ . M.p.  $186-7^\circ$ . Mod. sol.  $H_2O$ .

Di-Me ether: see 4-Nitro-2 : 3-dimethoxybenzoic acid.

Cain, Simonsen, Smith, *J. Chem. Soc.*, 1914, **105**, 1341.

## 5-Nitro-2 : 3-dihydroxybenzoic Acid.

3-Me ether: needles from  $H_2O$ . M.p.  $227^\circ$  decomp.  $FeCl_3 \rightarrow$  reddish-brown col.

Di-Me ether: see 5-Nitro-2 : 3-dimethoxybenzoic acid

3-Et ether:  $C_9H_9O_6N$ . MW, 227. Needles from EtOH. M.p.  $205^\circ$ . Spar. sol.  $H_2O$ . Alc.  $FeCl_3 \rightarrow$  brownish-violet col. rapidly turning brown.

2-Me-3-Et ether:  $C_{10}H_{11}O_6N$ . MW, 241. Needles from 90% EtOH. M.p.  $177^\circ$ . Spar. sol. cold  $H_2O$ . No col. with  $FeCl_3$ .

3-Me-2-Et ether: needles. M.p.  $169^\circ$ . Spar. sol. cold  $H_2O$ .

Di-Et ether:  $C_{11}H_{13}O_6N$ . MW, 255. Needles. M.p.  $118^\circ$ .

Klemenc, *Monatsh.*, 1914, **35**, 98.

Davies, *J. Chem. Soc.*, 1923, **123**, 1588.

Davies, Rubenstein, *J. Chem. Soc.*, 1923, **123**, 2848.

## 6-Nitro-2 : 3-dihydroxybenzoic Acid.

Di-Me ether: see 6-Nitro-2 : 3-dimethoxybenzoic acid.

3-Me-2-Et ether: cryst. from hot  $H_2O$ . M.p.  $119-20^\circ$ . Mod. sol. hot  $H_2O$ .

3-Me ether: 2-benzenesulphonyl, leaflets from EtOH.Aq. M.p.  $218^\circ$ .

Davies, *J. Chem. Soc.*, 1923, **123**, 1585.

Rubenstein, *J. Chem. Soc.*, 1925, 2269.

## Nitro-2 : 4-dihydroxybenzoic Acid.

See Nitro- $\beta$ -resorcylic Acid.

## Nitro-2 : 5-dihydroxybenzoic Acid.

See Nitrogentic Acid.

## Nitro-2 : 6-dihydroxybenzoic Acid.

See Nitro- $\gamma$ -resorcylic Acid.

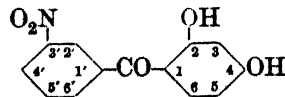
## Nitro-3 : 4-dihydroxybenzoic Acid.

See Nitroprotocatechuic Acid.

## Nitro-3 : 5-dihydroxybenzoic Acid.

See Nitro- $\alpha$ -resorcylic Acid.

## 3'-Nitro-2 : 4-dihydroxybenzophenone

 $C_{13}H_9O_5N$ 

MW, 259

Yellow needles from EtOH.Aq. M.p.  $228^\circ$ .

*Di-Me ether* :  $C_{15}H_{13}O_5N$ . MW, 287. Cryst. from EtOH. M.p. 116–17°.

Yamashita, *Bull. Chem. Soc. Japan*, 1928, 3, 180.

#### 4'-Nitro-2 : 4-dihydroxybenzophenone.

Pale yellow cryst. from EtOH.Aq. M.p. 203°. Sol. EtOH, AcOH. Less sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, hot H<sub>2</sub>O. FeCl<sub>3</sub> → reddish-brown col. KOH → dark red sol.

*Di-Me ether* : pale yellow cryst. from pet. ether. M.p. 123–4°.

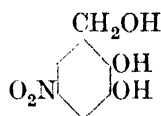
See previous reference.

#### 4'-Nitro-2 : 5-dihydroxybenzophenone.

*Di-Me ether* :  $C_{15}H_{13}O_5N$ . MW, 287. Yellow cryst. from EtOH. M.p. 126°. Sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Mod. sol. EtOH, AcOH, AcOEt, CS<sub>2</sub>. Spar. sol. Et<sub>2</sub>O, ligroin. Conc. H<sub>2</sub>SO<sub>4</sub> → intense red col. *Oxime* : exists in two forms. (i) Cryst. from EtOH. M.p. 195°. Sol. AcOH. Spar. sol. EtOH, CHCl<sub>3</sub>, Et<sub>2</sub>O, ligroin. In boiling toluene passes into second form. (ii) Cryst. M.p. 145°. More sol. than first form. *Phenylhydrazones* : exists in three forms. M.ps., 165°, 145° and 81°.

Kauffmann, de Pay, *Ber.*, 1912, 45, 778.

#### 5-Nitro-2 : 3-dihydroxybenzyl Alcohol



$C_9H_7O_5N$

MW, 185

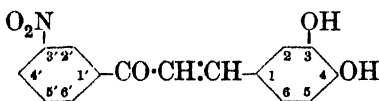
2 : 3-*Di-Me ether* :  $C_9H_{11}O_5N$ . MW, 213. Needles from MeOH. M.p. 67°.

3-*Me-2-Et ether* :  $C_{10}H_{13}O_5N$ . MW, 227. Pale yellow prisms from EtOH. M.p. 132°.

*Di-Et ether* :  $C_{11}H_{15}O_5N$ . MW, 241. Needles from EtOH. M.p. 75°.

Rubenstein, *J. Chem. Soc.*, 1926, 649.

#### 3'-Nitro-3 : 4-dihydroxychalkone



$C_{15}H_{11}O_5N$

MW, 285

Yellow needles from EtOH.Aq. M.p. 217°.

*Di-Et ether* :  $C_{19}H_{19}O_5N$ . MW, 341. Yellow needles from EtOH-Et<sub>2</sub>O. M.p. 103°.

*Diacetyl* : needles from EtOH. M.p. 179°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

Rupe, Wassergug, *Ber.*, 1901, 34, 3530.

#### 3-Nitro-4 : 4'-dihydroxychalkone.

*Di-Me ether* :  $C_{17}H_{15}O_5N$ . MW, 313. Yellow needles from EtOH. M.p. 160°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOH. Spar. sol. MeOH, EtOH. Insol. Et<sub>2</sub>O, ligroin. *Perchlorate* : m.p. 177–80°.

Pfeiffer, Segall, *Ann.*, 1928, 460, 130.

#### 2-Nitro-α : 3-dihydroxycinnamic Acid



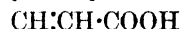
$C_9H_7O_6N$

MW, 225

*Di-Me ether* :  $C_{11}H_{11}O_6N$ . MW, 253. Needles from AcOH. M.p. 202° decomp. No col. with FeCl<sub>3</sub>. Alk. salts yellow.

Blaikie, Perkin, *J. Chem. Soc.*, 1924, 125, 334.

#### 5-Nitro-2 : 3-dihydroxycinnamic Acid



$C_9H_7O_6N$

MW, 225

*Di-Me ether* : prisms from EtOH. M.p. 231° (229°). Ox. → 5-nitro-*o*-veratric acid.

*Me ester* :  $C_{12}H_{13}O_6N$ . MW, 267. Needles from EtOH. M.p. 154–5°. *Et ester* :  $C_{13}H_{15}O_6N$ . MW, 281. Prisms from EtOH. M.p. 116° (111°).

2-*Me-3-Et ether* :  $C_{12}H_{13}O_6N$ . MW, 267. Needles from EtOH. M.p. 200–1°.

*Di-Et ether* :  $C_{13}H_{15}O_6N$ . MW, 281. Prisms from EtOH. M.p. 199°.

Rubenstein, *J. Chem. Soc.*, 1926, 651.

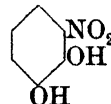
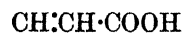
Chakravarti, Perkin, *J. Chem. Soc.*, 1929, 194.

#### 6-Nitro-2 : 3-dihydroxycinnamic Acid.

*Di-Me ether* : needles from EtOH.Aq. M.p. 210–15°. *Et ester* : needles from EtOH.Aq. M.p. 86°.

See first reference above.

#### 2-Nitro-3 : 4-dihydroxycinnamic Acid (2-Nitrocaffeic acid)



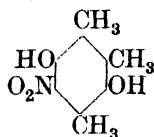
$C_9H_7O_6N$

MW, 225

*Trans* :

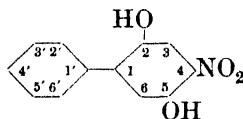
*Di-Me ether* : needles from EtOH. M.p. 229° after softening at 217°.

Gulland, Virden, *J. Chem. Soc.*, 1928, 932.

**5-Nitro-3 : 6-dihydroxy-ψ-cumene** (5-Nitro-3 : 6-dihydroxy-1 : 2 : 4-trimethylbenzene) $C_9H_{11}O_4N$ 

MW, 197

Golden-yellow needles from  $Et_2O$ . M.p. 106°. Sol.  $EtOH$ ,  $Et_2O$ ,  $CHCl_3$ ,  $AcOH$ . Less sol. hot  $H_2O$ .

Nef, *Ann.*, 1887, 237, 18.**4-Nitro-2 : 5-dihydroxydiphenyl** $C_{12}H_9O_4N$ 

MW, 231

*Di-Me ether* :  $C_{14}H_{13}O_4N$ . MW, 259. M.p. 102-3°.

*Di-Et ether* :  $C_{16}H_{17}O_4N$ . MW, 287. M.p. 92°.

*Dibutyl ether* :  $C_{20}H_{25}O_4N$ . MW, 343. M.p. 41-2°.

*Dibenzyl ether* :  $C_{26}H_{21}O_4N$ . MW, 411. M.p. 136-8°.

I.G., F.P., 739,053, (*Chem. Abstracts*, 1933, 27, 1893); D.R.P., 566,521, (*Chem. Abstracts*, 1933, 27, 2459).

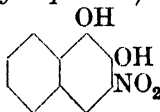
**4-Nitro-2 : 4'-dihydroxydiphenyl.**

Yellow cryst. M.p. 187°. Sol. alkalis and alk. carbonates.

Finzi, Mangini, *Gazz. chim. ital.*, 1932, 62, 673.

**5-Nitro-3 : 6-dihydroxy-2-methylbenzoquinone.**

See 5-Nitro-3 : 6-dihydroxytoluquinone.

**3-Nitro-1 : 2-dihydroxynaphthalene** (3-Nitro-β-naphthohydroquinone) $C_{10}H_7O_4N$ 

MW, 205

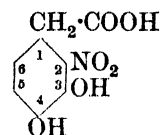
Red plates from  $EtOH$  or  $C_6H_6$ , needles from  $H_2O$ . M.p. 159-5° (152-3°). Sol.  $EtOH$ ,  $AcOH$ . Spar. sol.  $H_2O$ . Sublimes in red needles.

*Diacetyl* : orange leaflets. M.p. 196-7°.

Zincke, Noack, *Ann.*, 1897, 295, 12.

**Nitro-9 : 10-dihydroxyphenanthrene.**

See Nitrophenanthrahydroquinone.

**2-Nitro-3 : 4-dihydroxyphenylacetic Acid** $C_8H_7O_6N$ 

MW, 213

Orange-yellow needles from xylene. M.p. 171°. Sol.  $EtOH$ . Sol.  $NaOH$  with orange col.  $FeCl_3$  → intense green col.

*3-Me ether* : see 2-Nitrohomovanillic Acid.

*3 : 4-Dibenzyl ether* :  $C_{22}H_{19}O_6N$ . MW, 393. Needles from  $EtOH$ . Aq., m.p. 85°.

*3 : 4-Dicarboxyl* : needles from  $C_6H_6$ , m.p. 115°; needles from  $AcOH$ , m.p. 127°.

*Di-Me ether* : see 2-Nitrohomoveratric Acid.

Gulland, *J. Chem. Soc.*, 1931, 2872.

**6-Nitro-3 : 4-dihydroxyphenylacetic Acid.**

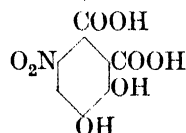
Yellow needles from toluene. M.p. 212°. Sol.  $H_2O$ .  $NaOH$  → cherry-red sol.  $FeCl_3$  → dark green col.

*Anilide* : needles from  $EtOH$ . M.p. 201-2°.

*3-Me ether* : see 6-Nitrohomovanillic Acid.

*Di-Me ether* : see 6-Nitrohomoveratric Acid.

See previous reference.

**6-Nitro-3 : 4-dihydroxyphthalic Acid** (6-Nitronorhemipinic acid) $C_8H_5O_8N$ 

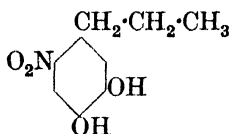
MW, 243

*4-Me ether* :  $C_9H_7O_8N$ . MW, 257. Needles from  $EtOH$ , leaflets from  $AcOEt$ -pet. ether. M.p. 220° (205-6° decomp.). Sol.  $H_2O$ ,  $EtOH$ ,  $AcOEt$ . Spar. sol.  $C_6H_6$ . *Di-Me ester* :  $C_{11}H_{11}O_8N$ . MW, 285. Leaflets from  $C_6H_6$ . M.p. 145-6°. Sol. hot  $H_2O$ .  $KOH$  → yellow sol. *2-Me ester-1-anilide* :  $C_{17}H_{16}O_7N_2$ . MW, 360. Cryst. from  $C_6H_6$ . M.p. 170° decomp. *1-Anilide* :  $C_{16}H_{14}O_7N_2$ . MW, 346. Cryst. powder from  $Me_2CO-CCl_4$ . M.p. 192°. Mod. sol.  $Me_2CO$ . Spar. sol.  $H_2O$ ,  $AcOEt$ .  $FeCl_3$  → ruby-red col. *2-Anilide* : pale yellow prisms from  $Me_2CO-CCl_4$ . M.p. 183-4° decomp. Sol.  $EtOH$ ,  $Me_2CO$ . Spar. sol.  $H_2O$ ,  $Et_2O$ ,  $C_6H_6$ ,  $CCl_4$ . Sol. alkalis and  $NH_3$ . Aq. with blood-red col.

*Di-Me ether* : see 6-Nitrohemipinic Acid.

Elbel, *Ber.*, 1886, 19, 2312.

Wegscheider, Klemenc, *Monatsh.*, 1911, 32, 386.

**6-Nitro-3 : 4-dihydroxy-1-propylbenzene****6-Nitro-3 : 4-dihydroxy-1-propylbenzene** $C_9H_{11}O_4N$ 

MW, 197

Yellowish-green cryst. M.p. 73°. Very sol.  $H_2O$  and most org. solvents.

3-*Me ether* :  $C_{10}H_{13}O_4N$ . MW, 211. Cryst. +  $H_2O$ . M.p. 78°.

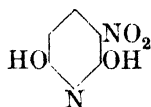
4-*Me ether* : cryst. M.p. 52°. 3-*Et ether* :  $C_{12}H_{17}O_4N$ . MW, 239. Yellow needles. M.p. 60°.

*Di-Me ether* :  $C_{11}H_{15}O_4N$ . MW, 225. Yellowish prisms. M.p. 81-2°.

Thoms, Biltz, *Arch. Pharm.*, 1904, **242**, 88.

**Nitro- $\alpha$ -dihydroxypropylbenzene.**

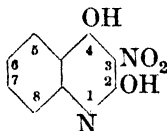
See 1-*o*-Nitrophenyltrimethylene Glycol.

**3-Nitro-2 : 6-dihydroxypyridine** $C_5H_4O_4N_2$ 

MW, 156

Yellow needles from  $H_2O$ . Decomp. at 321°. Sol. alkalis with yellow col.

Gattermann, Skita, *Ber.*, 1916, **49**, 499.

**3-Nitro-2 : 4-dihydroxyquinoline** $C_9H_6O_4N_2$ 

MW, 206

Yellow prisms from AcOH. Decomp. at 225°. Sol. alkalis and  $NH_3$ . Aq.

*Acetyl* : yellow cryst. M.p. 194° decomp.

Gabriel, *Ber.*, 1918, **51**, 1500.

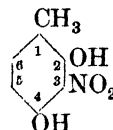
Ashley, Perkin, Robinson, *J. Chem. Soc.*, 1930, 382.

**8-Nitro-5 : 6-dihydroxyquinoline.**

*Di-Me ether* :  $C_{11}H_{10}O_4N_2$ . MW, 234. Cryst. M.p. 126-8°.

I.G., D.R.P., 531,083, (*Chem. Abstracts*, 1931, **25**, 5434).

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**2-Nitro-4' : 4''-dihydroxytriphenylmethane****3-Nitro-2 : 4-dihydroxytoluene (3-Nitro-cresorcinol)** $C_7H_7O_4N$ 

MW 169

Red cryst. from EtOH. M.p. 112°. Sol. EtOH,  $C_6H_6$ , AcOH, pet. ether. Sol. cold  $Na_2CO_3$ . Aq. Volatile in steam.

Henrich, Fleischmann, *Ber.*, 1930, **63**, 1337.

**5-Nitro-2 : 4-dihydroxytoluene (5-Nitro-cresorcinol).**

Orange-red needles from  $C_6H_6$  or ligroin. M.p. 125°. Sol. cold  $Na_2CO_3$ . Aq. Non-volatile in steam.

See previous reference.

**6-Nitro-2 : 5-dihydroxytoluene.**

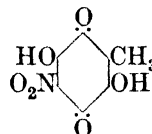
See 6-Nitrotoluidhydroquinone.

**Nitro-3 : 4-dihydroxytoluene.**

See Nitrohomocatechol.

**Nitro-3 : 5-dihydroxytoluene.**

See Nitro-oreinol.

**5-Nitro-3 : 6-dihydroxytoluquinone (5-Nitro-3 : 6-dihydroxy-2-methylbenzoquinone, tolu-nitrannilic acid)** $C_7H_5O_6N$ 

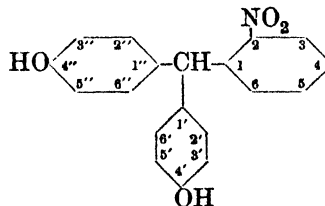
MW, 199

Yellow needles +  $H_2O$  from  $H_2O$ . M.p. 180° decomp.

Kehrmann, Brasch, *J. prakt. Chem.*, 1889, **39**, 378.

**Nitrodihydroxytrimethylbenzene.**

See Nitrodihydroxy-*p*-cumene.

**2 - Nitro - 4' : 4'' - dihydroxytriphenyl - methane** $C_{19}H_{15}O_4N$ 

MW, 321

Brown amorph. powder. M.p. 92°. Sol. EtOH,  $Et_2O$ , AcOH,  $Me_2CO$ , AcOEt. Spar.

**3-Nitro-4' : 4''-dihydroxytriphenyl-methane**

sol.  $C_6H_6$ . Alkalis  $\rightarrow$  red sols.  $\rightarrow$  intense green fluor. with EtOH. Conc.  $H_2SO_4 \rightarrow$  intense red sol.

*Di-Me ether* :  $C_{21}H_{19}O_4N$ . MW, 349. Brown amorph. powder.

*Diacetyl* : yellow amorph. powder.

*Dibenzoyl* : yellowish powder from 96% EtOH. M.p.  $155^\circ$ .  $C_6H_6 \rightarrow$  reddish-brown sol. with green fluor.

Tănăsescu, Simonescu, *J. prakt. Chem.*, 1934, 141, 319.

**3 - Nitro - 4' : 4'' - dihydroxytriphenyl - methane.**

Reddish powder. M.p.  $90-1^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $AcOEt$ ,  $AcOH$ . Less sol.  $C_6H_6$ . Alkalis  $\rightarrow$  yellow sols.  $\rightarrow$  green fluor. with EtOH.

*Di-Me ether* : yellow amorph. powder from MeOH. M.p.  $70^\circ$ .

*Dibenzoyl* : amorph. powder from 96% EtOH. M.p.  $89^\circ$ .

See previous reference.

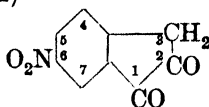
**4 - Nitro - 4' : 4'' - dihydroxytriphenyl - methane.**

Yellowish amorph. powder. M.p.  $130^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $Et_2O$ ,  $AcOH$ . Less sol.  $C_6H_6$ , xylene. Conc.  $H_2SO_4 \rightarrow$  red sol.

*Di-Me ether* : reddish amorph. powder. Sol.  $Et_2O$ .

*Dibenzoyl* : powder. Sol.  $C_6H_6$ . Less sol. 96% EtOH.

See previous reference.

**6-Nitro-1 : 2-diketohydrindene (6-Nitro-indandione-1 : 2)**

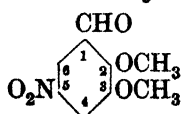
$C_9H_5O_4N$  MW, 191

Pale orange-brown needles. Decomp. about  $150^\circ$ . Sol. alkalis with reddish-brown col.

*2-Oxime* : plates from EtOH, prisms from EtOH. M.p. about  $240^\circ$  decomp. ( $196^\circ$ ). Mod. sol.  $AcOH$ . Spar. sol. EtOH. Alkalis  $\rightarrow$  deep purple sols. which rapidly turn brown.

Ingold, Piggott, *J. Chem. Soc.*, 1923, 123, 1488.

v. Braun, Heider, *Ber.*, 1916, 49, 1279.

**5-Nitro-2 : 3-dimethoxybenzaldehyde**

$C_9H_5O_5N$

MW, 211  $C_9H_5O_5N$

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**3-Nitro-2 : 5-dimethoxybenzaldehyde**

Needles from MeOH. M.p.  $115^\circ$  ( $116-17^\circ$ ). Very sol. hot MeOH. Spar. sol. boiling  $H_2O$ .

*Di-Me acetal* : prisms from MeOH. M.p.  $89^\circ$  ( $96-7^\circ$ ).

*Oxime* : needles from EtOH. M.p.  $155^\circ$ .

*Semicarbazone* : needles from EtOH. Aq. M.p.  $210^\circ$ .

*Phenylhydrazone* : red plates from EtOH. M.p.  $179^\circ$ .

*Anil* : needles from EtOH. M.p.  $122^\circ$ .

*p-Me-anil* : needles from EtOH. M.p.  $143^\circ$ .

Perkin, Robinson, Stoyale, *J. Chem. Soc.*, 1924, 125, 2358.

Davies, *J. Chem. Soc.*, 1923, 123, 1580.

**6-Nitro-2 : 3-dimethoxybenzaldehyde.**

Needles from  $C_6H_6$ -ligroin. M.p.  $110^\circ$ .

*Di-Me acetal* : cubes from MeOH. M.p.  $72^\circ$ . Turns green in air.

*Oxime* : needles from EtOH. Aq. M.p.  $130^\circ$ .

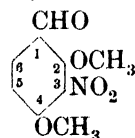
*Semicarbazone* : needles from EtOH. Aq. M.p.  $227-8^\circ$ .

*Phenylhydrazone* : brownish-orange prisms from EtOH. M.p.  $138^\circ$ .

*Anil* : needles from EtOH. M.p.  $84^\circ$ .

*p-Me-anil* : plates from EtOH. M.p.  $104^\circ$ .

Perkin, Robinson, Stoyale, *J. Chem. Soc.*, 1924, 125, 2358.

**3-Nitro-2 : 4-dimethoxybenzaldehyde (3-Nitroresorcylic aldehyde dimethyl ether)**

$C_9H_9O_5N$  MW, 211

Cryst. M.p.  $104-5^\circ$ .

Srikantia, Iyengar, Santanam, *J. Chem. Soc.*, 1932, 527.

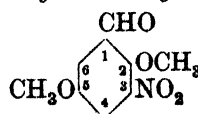
**5-Nitro-2 : 4-dimethoxybenzaldehyde (5-Nitroresorcylic aldehyde dimethyl ether).**

Needles from MeOH. M.p.  $188-9^\circ$ . Spar. sol. alcohols. No col. with  $FeCl_3$ .

*Oxime* : pale yellow. M.p.  $184-5^\circ$ .

*Hydrazone* : orange plates. M.p.  $169-70^\circ$ .

Rao, Srikantia, Iyengar, *J. Chem. Soc.*, 1925, 127, 558.

**3-Nitro-2 : 5-dimethoxybenzaldehyde (3-Nitroresorcylic aldehyde dimethyl ether)**

MW, 211

Pale yellow needles from  $\text{CHCl}_3$ . M.p.  $113^\circ$ .

Rubenstein, *J. Chem. Soc.*, 1925, 127, 2000.

**6-Nitro-2 : 5-dimethoxybenzaldehyde** (6-Nitrogentisic aldehyde dimethyl ether).

Pale yellow needles from EtOH. M.p.  $159^\circ$ .

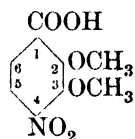
p-Nitrophenylhydrazone : scarlet prisms. M.p.  $245-50^\circ$  decomp.

See previous reference.

**Nitro-3 : 4-dimethoxybenzaldehyde.**

See Nitroveratric Aldehyde.

**4-Nitro-2 : 3-dimethoxybenzoic Acid**



$\text{C}_9\text{H}_9\text{O}_6\text{N}$

MW, 227

Two compounds of this structure have been described in the literature.

1. Pale yellow needles from  $\text{H}_2\text{O}$ . M.p.  $94-5^\circ$ .

Majima, Okazaki, *Ber.*, 1916, 49, 1494.

2. Prisms from AcOEt. M.p.  $215-16^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ .

*Et ester* :  $\text{C}_{11}\text{H}_{13}\text{O}_6\text{N}$ . MW, 255. Needles from EtOH. M.p.  $142-3^\circ$ .

Cain, Simonsen, Smith, *J. Chem. Soc.*, 1914, 105, 1339.

**5-Nitro-2 : 3-dimethoxybenzoic Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $176^\circ$  ( $174-5^\circ$ ).

*Me ester* :  $\text{C}_{10}\text{H}_{11}\text{O}_6\text{N}$ . MW, 241. Needles from MeOH.Aq. M.p.  $76-7^\circ$ .

*Et ester* : needles from EtOH. M.p.  $79^\circ$ .

*Nitrile* :  $\text{C}_9\text{H}_8\text{O}_4\text{N}_2$ . MW, 208. Brownish needles from MeOH.Aq. M.p. about  $127-8^\circ$ . Sol. most org. solvents.

Gibson, Simonsen, Rau, *J. Chem. Soc.*, 1917, 111, 77.

**6-Nitro-2 : 3-dimethoxybenzoic Acid.**

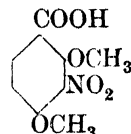
Leaflets or needles from  $\text{H}_2\text{O}$ . M.p.  $189-90^\circ$  ( $184-5^\circ$ ). Mod. sol. hot  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

*Me ester* : needles from MeOH. M.p.  $81^\circ$ .

Perkin, Robinson, *J. Chem. Soc.*, 1914, 105, 2390.

Cain, Simonsen, *ibid.*, 161.

**3-Nitro-2 : 4-dimethoxybenzoic Acid** (3-Nitro- $\beta$ -resorcylic acid dimethyl ether)



$\text{C}_9\text{H}_9\text{O}_6\text{N}$

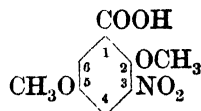
MW, 227

Needles from  $\text{H}_2\text{O}$ . M.p.  $210-12^\circ$ .

*Me ester* :  $\text{C}_{10}\text{H}_{11}\text{O}_6\text{N}$ . MW, 241. Prisms from MeOH. M.p.  $118-19^\circ$ .

Dodswell, Kenner, *J. Chem. Soc.*, 1927, 587.

**3-Nitro-2 : 5-dimethoxybenzoic Acid** (3-Nitrogentisic acid dimethyl ether)



$\text{C}_9\text{H}_9\text{O}_6\text{N}$

MW, 227

Yellowish needles from  $\text{H}_2\text{O}$ . M.p.  $181-3^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ . Alkalis  $\rightarrow$  yellow sols.

*Me ester* :  $\text{C}_{10}\text{H}_{11}\text{O}_6\text{N}$ . MW, 241. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $71-2^\circ$ .

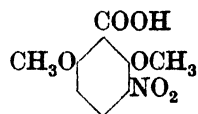
Klemenc, *Monatsh.*, 1912, 33, 1253.

**6-Nitro-2 : 5-dimethoxybenzoic Acid** (6-Nitrogentisic acid dimethyl ether).

*Me ester* : yellow cryst. powder from  $\text{C}_6\text{H}_6$ -pet. ether. M.p.  $117-18^\circ$ .

Klemenc, *Monatsh.*, 1914, 35, 105.

**3-Nitro-2 : 6-dimethoxybenzoic Acid** (3-Nitro- $\gamma$ -resorcylic acid dimethyl ether)



$\text{C}_9\text{H}_9\text{O}_6\text{N}$

MW, 227

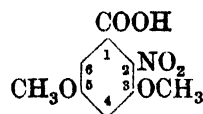
*Nitrile* :  $\text{C}_9\text{H}_8\text{O}_4\text{N}_2$ . MW, 208. Yellow needles from EtOH. M.p.  $112^\circ$ .

Blanksma, *Chem. Zentr.*, 1912, II, 339.

**Nitro-3 : 4-dimethoxybenzoic Acid.**

See Nitroveratric Acid.

**2-Nitro-3 : 5-dimethoxybenzoic Acid** (2-Nitro- $\alpha$ -resorcylic acid dimethyl ether)



$\text{C}_9\text{H}_9\text{O}_6\text{N}$

MW, 227



Cryst. M.p. 232°. Turns yellow in air.

*Et ester*:  $C_{11}H_{13}O_6N$ . MW, 255. Needles. M.p. 131°.

*Amide*:  $C_9H_{10}O_5N_2$ . MW, 226. Pale yellowish leaflets from EtOH. M.p. 199°.

I.G., D.R.P., 501,609, (*Chem. Zentr.*, 1930, II, 1773).

**4-Nitro-3 : 5-dimethoxybenzoic Acid (4-Nitro- $\alpha$ -resorcylic acid dimethyl ether).**

Yellow needles from  $H_2O$ , prisms from EtOH or AcOH. M.p. 225°. Sol. hot EtOH, AcOH. Spar. sol.  $H_2O$ . Sublimes in needles with part. decomp.

*Et ester*: pale yellow needles from EtOH or  $Et_2O$ . M.p. 130°.

Einhorn, Pfyl, *Ann.*, 1900, **311**, 62.

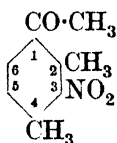
**Nitro-3 : 4-dimethoxyphenylacetic Acid.**

See Nitrohomoveratric Acid.

**Nitro-3 : 4-dimethoxytoluene.**

See Nitrohomoveratrol.

**3-Nitro-2 : 4-dimethylacetophenone**



$C_{10}H_{11}O_3N$

MW, 193

Prisms from AcOH. M.p. 72°.

Claus, *J. prakt. Chem.*, 1890, **41**, 499.

**5-Nitro-2 : 4-dimethylacetophenone.**

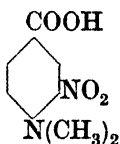
Needles from EtOH. M.p. 67°. Very sol. hot EtOH. Sublimes in needles.

See previous reference.

***N*-Nitrodimethylamine.**

See Dimethylnitramine.

**3-Nitro-*p*-dimethylaminobenzoic Acid**



$C_9H_{10}O_4N_2$

MW, 210

Golden-yellow needles from toluene or  $CHCl_3$ . M.p. 222-3° (213°). Very sol. EtOH. Insol.  $H_2O$ .

*Me ester*:  $C_{10}H_{12}O_4N_2$ . MW, 224. Yellow leaflets from EtOH.Aq. M.p. 71.5°. Very sol. EtOH,  $Me_2CO$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Mod. sol. ligroin. Spar. sol.  $H_2O$ .

*Et ester*:  $C_{11}H_{14}O_4N_2$ . MW, 238. Pale yellow leaflets from EtOH. M.p. 80-1°.

*Amide*:  $C_9H_{11}O_3N_3$ . MW, 209. Cryst. from  $H_2O$ . M.p. 210°. Sol.  $CHCl_3$ . Mod. sol. EtOH. Insol.  $Et_2O$ , ligroin.

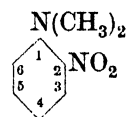
*Nitrile*:  $C_9H_9O_2N_3$ . MW, 191. Yellow plates. M.p. 114-15°. Mod. sol. EtOH,  $C_6H_6$ .

Noelting, Demant, *Ber.*, 1904, **37**, 1030.

Reverdin, Delétra, *Ber.*, 1906, **39**, 972.

Reverdin, *Ber.*, 1907, **40**, 2443.

***o*-Nitro-*N*-dimethylaniline**



$C_8H_{10}O_2N_2$

MW, 166

Orange-yellow oil. F.p. -20°. B.p. 151-3°/30-3 mm.  $n_D$  1.61021. Mod. sol.  $H_2O$ . Easily volatile in steam.

*B, HCl*: needles. M.p. 173-4°. Sol.  $H_2O$ , hot EtOH,  $CHCl_3$ .

*B, HBr*: prisms from EtOH. Decomp. at 172°.

*B, HI*: cryst. Decomp. at 126°. Unstable.

*B, H\_2SO\_4*: plates from EtOH. M.p. 168° (126-7°).

*B, HAuCl\_4*: yellowish prisms. Decomp. at 152°. Spar. sol. EtOH.

*Picrate*: pale yellow prisms from EtOH. M.p. 102-103.5°. Very sol.  $Me_2CO$ . Sol. hot EtOH,  $Et_2O$ .

$C_8H_{10}O_2N_2, C_6H_3(NO_2)_3 \cdot 1 : 3 : 5$ : yellowish-red cryst. M.p. 112°.

Friedländer, *Monatsh.*, 1898, **19**, 636.

Weissenberger, *Monatsh.*, 1912, **33**, 826.

***m*-Nitro-*N*-dimethylaniline.**

Red cryst. from  $Et_2O$  or EtOH- $Et_2O$ . M.p. 60-1°. B.p. 280-5°. Volatile in steam.

*Methiodide*: plates from  $H_2O$ .

*Methonitrate*: prisms or plates from  $H_2O$ . M.p. 220-40° decomp.

*Methopicrate*: yellow prisms from EtOH.Aq. M.p. 151-3°.

*Picrate*: yellow cryst. M.p. 119°.

Ullmann, *Ann.*, 1903, **327**, 112.

Vorländer, Siebert, *Ber.*, 1919, **52**, 294.

***p*-Nitro-*N*-dimethylaniline.**

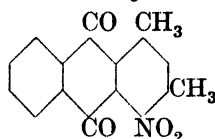
Yellow needles with blue reflex from EtOH. M.p. 163°. Sol. warm EtOH. AcOH. Insol.  $H_2O$ .

*B, 2HCl*: m.p. about 53°.

Tingle, Blanck, *J. Am. Chem. Soc.*, 1908, **30**, 1405.

Davies, *Bull. soc. chim.*, 1935, **2**, 295.

## 4-Nitro-1 : 3-dimethylantraquinone

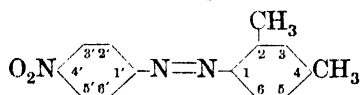
 $C_{16}H_{11}O_4N$ 

MW, 281

Pale yellow needles from AcOH. M.p. 234°. Turns orange-yellow in air.

Scholl, *Ber.*, 1910, **43**, 353.

## 4'-Nitro-2 : 4-dimethylazobenzene

 $C_{14}H_{13}O_2N_3$ 

MW, 255

Orange-red needles from EtOH. M.p. 128-5-129-5°. Sol. hot  $Me_2CO$ ,  $C_6H_6$ , hot ligroin.

Bamberger, *Ber.*, 1907, **40**, 1913, 1923, 1932.

## 4'-Nitro-3 : 4-dimethylazobenzene.

Orange-red needles. M.p. 135-5°.

Bamberger, Blangey, *Ann.*, 1911, **384**, 318.

## 6-Nitro-2 : 4'-dimethylazobenzene.

Red prisms from AcOH. M.p. 65-5-66°. Sol. EtOH,  $Et_2O$ , AcOH.

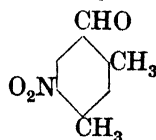
Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1172.

## 2-Nitro-4 : 4'-dimethylazobenzene.

Orange-red cryst. from EtOH. M.p. 80°.  $Sn + HCl \rightarrow p$ -toluidine and 3 : 4-diaminotoluene.

Janovsky, *Monatsh.*, 1889, **10**, 586.

## 5-Nitro-2 : 4-dimethylbenzaldehyde

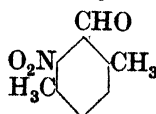
 $C_9H_9O_3N$ 

MW, 179

Yellowish needles from ligroin. M.p. 81°.

Gattermann, *Ann.*, 1906, **347**, 372.

## 6-Nitro-2 : 5-dimethylbenzaldehyde

 $C_9H_9O_3N$ 

MW, 179

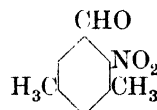
Yellow needles or leaflets from EtOH. M.p. 120°.

Semicarbazone : needles from EtOH. M.p. 183°.

Azine : yellowish needles from EtOH. M.p. 162°.

Gattermann, *Ann.*, 1912, **393**, 221.

## 2-Nitro-3 : 5-dimethylbenzaldehyde

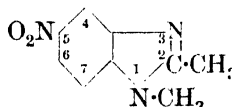
 $C_9H_9O_3N$ 

MW, 179

Needles. M.p. 102-3°. Sol. EtOH, pet. ether.

Bamberger, Weiler, *J. prakt. Chem.*, 1898, **58**, 360.

## 5-Nitro-1 : 2-dimethylbenziminazole

 $C_9H_9O_2N_3$ 

MW, 191

Yellow plates from EtOH or  $PhNO_2$ . M.p. 226°.

Freis, Modrow, Racke, Weber, *Ann.*, 1927, **454**, 219.

## 6-Nitro-1 : 2-dimethylbenziminazole.

Cryst. from 90% EtOH. M.p. 242°.

Phillips, *J. Chem. Soc.*, 1931, 1151.

## 6-Nitro-2 : 5-dimethylbenziminazole.

Needles +  $H_2O$  from  $H_2O$ . M.p. 201-2°. Very sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Sol. hot  $H_2O$ .

Maron, Salzberg, *Ber.*, 1911, **44**, 2999.

Niementowsky, *Ber.*, 1896, **19**, 724.

Ladenburg, *Ber.*, 1875, **8**, 677.

## 7-Nitro-2 : 5-dimethylbenziminazole.

Needles. M.p. 246°. Sol. EtOH. Spar. sol.  $Et_2O$ .

Bankiewicz, *Ber.*, 1888, **21**, 2402.

## 5-Nitro-2 : 7-dimethylbenziminazole.

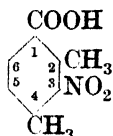
Cryst. from hot  $H_2O$ . M.p. 186°. Sol. EtOH. Spar. sol. cold  $H_2O$ . Sol. dil. HCl. Cold dil. NaOH  $\rightarrow$  yellow sol.

Kym, Ringer, *Ber.*, 1915, **48**, 1675.

## 6-Nitro-4 : 7-dimethylbenziminazole.

Pale yellow needles from 50% EtOH. M.p. 221°. Sol. EtOH, AcOH. Spar. sol.  $C_6H_6$ , pet. ether. Sol. bases with yellow col.

Fries, Noll, *Ann.*, 1912, **389**, 375.

**3-Nitro-2 : 4-dimethylbenzoic Acid** (3-Nitro-unsym.-m-xylylic acid) $C_9H_9O_4N$ 

MW, 195

Yellow prisms from EtOH. M.p. 179° (135°). Sol. EtOH. Spar. sol. hot  $H_2O$ ,  $C_6H_6$ .

Amide :  $C_9H_{10}O_3N_2$ . MW, 194. Plates from  $H_2O$ . M.p. 138°.

Nitrile :  $C_9H_8O_2N_2$ . MW, 176. M.p. 120-1°.

Wheeler, Hoffman, *Am. Chem. J.*, 1911, 45, 441.

Borsche, *Ann.*, 1912, 386, 366.

**5-Nitro-2 : 4-dimethylbenzoic Acid.**

Needles from  $H_2O$ . M.p. 197.5-198.5°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ . Spar. sol.  $H_2O$ .

Et ester :  $C_{11}H_{13}O_4N$ . MW, 223. Needles from EtOH.Aq. M.p. 75-6°.

Amide : m.p. 183°.

Nitrile :  $C_9H_8O_2N_2$ . MW, 176. Needles from EtOH. M.p. 108-9°.

Fisher, Walling, *J. Am. Chem. Soc.*, 1935, 57, 1701.

Ahrens, *Ann.*, 1892, 271, 18.

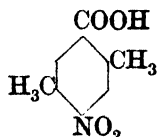
Borsche, *Ann.*, 1912, 386, 366.

**6-Nitro-2 : 4-dimethylbenzoic Acid.**

Yellow needles. M.p. 180°. Sol. EtOH. Spar. sol.  $H_2O$ .

Nitrile : yellow needles from  $H_2O$  or EtOH. M.p. 126°. Volatile in steam.

Kalle, D.R.P., 239,092, (*Chem. Zentr.*, 1911, II, 1292).

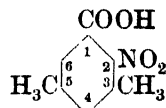
**4-Nitro-2 : 5-dimethylbenzoic Acid** $C_9H_9O_4N$ 

MW, 195

Cryst. from ligroin. M.p. 165.5-166.5°.

Nitrile : cryst. from EtOH. M.p. 160-1°. Sublimes.

Fisher, Walling, *J. Am. Chem. Soc.*, 1935, 57, 1702.

**2-Nitro-3 : 5-dimethylbenzoic Acid** (2-Nitro-sym.-m-xylylic acid, 2-nitromesitylenic acid) $C_9H_9O_4N$ 

MW, 195

Needles from hot  $H_2O$ . M.p. 210-12°. Sol. hot  $H_2O$ . Spar. sol. cold  $H_2O$ .

Et ester :  $C_{11}H_{13}O_4N$ . MW, 223. Plates from EtOH. M.p. 65-6°. Sol. EtOH. Insol.  $H_2O$ .

Schmitz, *Ann.*, 1878, 193, 162, 167.

**4-Nitro-3 : 5-dimethylbenzoic Acid** (4-Nitro-sym.-m-xylylic acid, 4-nitromesitylenic acid).

Prisms from  $H_2O$  or dil. alc. sol. M.p. 179° (174-6°). Cryst. from conc. alc. sol. M.p. 214-20° (223°); latter on recryst. from  $H_2O$  —> former (m.p. 179°).

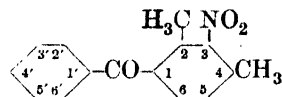
Et ester : prisms from EtOH. M.p. 72°. Sol. EtOH. Insol.  $H_2O$ .

Jacobsen, *Ber.*, 1878, 11, 2054.

Fittig, Brueckner, *Ann.*, 1868, 147, 48.

Wheeler, Hoffman, *Am. Chem. J.*, 1910, 44, 119.

Schmitz, *Ann.*, 1878, 193, 162, 167.

**3-Nitro-2 : 4-dimethylbenzophenone** $C_{15}H_{13}O_3N$ 

MW, 255

Brown prisms from MeOH. M.p. 79.5-80°. Sol. usual org. solvents. Spar. sol.  $H_2O$ .

Chardonnens, *Helv. Chim. Acta*, 1929, 12, 659.

**5-Nitro-2 : 4-dimethylbenzophenone.**

Yellowish prisms from MeOH. M.p. 62-3°. Sol. usual org. solvents. Spar. sol.  $H_2O$ .

See previous reference.

**3'-Nitro-2 : 4-dimethylbenzophenone.**

Yellowish leaflets from EtOH. M.p. 64°. Sol. hot EtOH,  $Me_2CO$ ,  $C_6H_6$ , AcOH,  $CS_2$ .

Oxime : cryst. from ligroin. M.p. 131-49°. Sol.  $Me_2CO$ ,  $Et_2O$ ,  $CHCl_3$ . Less sol. EtOH,  $C_6H_6$ ,  $CS_2$ . Spar. sol. ligroin.

Limpricht, Falkenberg, *Ann.*, 1895, 286, 333.

**3'-Nitro-2 : 5-dimethylbenzophenone.**

Needles from EtOH. M.p. 97-8°.

Limpricht, Falkenberg, *Ann.*, 1895, 286, 341.

**3'-Nitro-3 : 4-dimethylbenzophenone.**

Yellow plates from EtOH. M.p. 100°. Sol. neutral solvents.

Limpricht, Falkenberg, *Ann.*, 1895, **286**, 339.

**Nitrodimethylcarbostyryl.**

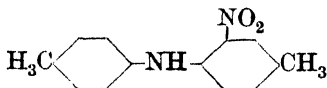
See Nitrohydroxydimethylquinoline.

**Nitrodimethyldiphenyl.**

See Nitroditolyl.

**Nitrodimethyldiphenyl Ether.**

See Nitroditolyl Ether.

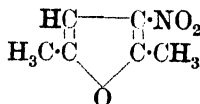
**2-Nitro-4 : 4'-dimethyldiphenylamine**

$C_{14}H_{14}O_2N_2$

MW, 242

Red plates from EtOH. M.p. 85°. Sol. most org. solvents.

Lellmann, *Ber.*, 1882, **15**, 831.

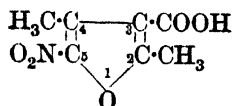
**3-Nitro-2 : 5-dimethylfuran**

$C_6H_7O_3N$

MW, 141

Liq. B.p. 105–10°/22 mm., 88–92°/9 mm.  $D^{20}_D$  1.25.  $n^{20}_D$  1.3140.

Gilman, Burtner, *Rec. trav. chim.*, 1932, **51**, 670.

**5-Nitro-2 : 4-dimethyl-β-furoic Acid (5-Nitro-2 : 4-dimethylfuran-3-carboxylic acid)**

$C_7H_7O_5N$

MW, 185

Cryst. M.p. 182°.

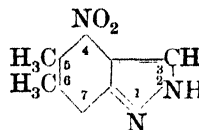
Gilman, Burtner, *Rec. trav. chim.*, 1932, **51**, 667.

**4-Nitro-2 : 5-dimethyl-β-furoic Acid (4-Nitro-2 : 5-dimethylfuran-3-carboxylic acid).**

Cryst. M.p. 176°.

*Et ester*:  $C_9H_{11}O_5N$ . MW, 213. Liq. B.p. 119–20°/20 mm.

See previous reference.

**4-Nitro-5 : 6-dimethylindazole**

$C_9H_9O_2N_3$

MW, 191

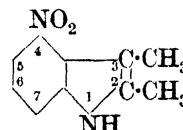
Leaflets from  $C_6H_6$ -ligroin. M.p. 204°. Sol. hot  $C_6H_6$ . Insol.  $H_2O$ , ligroin. Sol. alkalis with dark yellow col.

Noelting, *Ber.*, 1904, **37**, 2596.

**7-Nitro-5 : 6-dimethylindazole.**

Pale yellow needles from  $C_6H_6$ . M.p. 180.5–181.5°. Sol. conc. alkalis with orange col.

See previous reference.

**4-(or 6-)Nitro-2 : 3-dimethylindole**

$C_{10}H_{10}O_2N_2$

MW, 190

Orange-red needles from ligroin, m.p. 126°; orange prisms from EtOH, m.p. 142°.

*N-Acetyl*: pale yellow needles from EtOH. M.p. 170°.

Bauer, Strauss, *Ber.*, 1932, **65**, 313.

Plant, Tomlinson, *J. Chem. Soc.*, 1933, 958.

**5-Nitro-2 : 3-dimethylindole.**

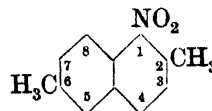
Orange prisms from EtOH, yellow prisms from ligroin. M.p. 188–9° (186°).

See previous references.

**7-Nitro-2 : 3-dimethylindole.**

Golden-yellow leaflets from ligroin. M.p. 164°.

Bauer, Strauss, *Ber.*, 1932, **65**, 313.

**1-Nitro-2 : 6-dimethylnaphthalene**

$C_{12}H_{11}O_2N$

MW, 201

Yellow leaflets from AcOH. M.p. 68°.

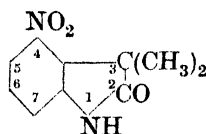
Mayer, Alken, *Ber.*, 1922, **55**, 2278.

**4-Nitro-2 : 6-dimethylnaphthalene.**

Yellow needles from MeOH. M.p. 84–5°.

Vesely, Štursa, *Chem. Zentr.*, 1933, **I**, 3078.

## 4-(or 6-)Nitro-3 : 3-dimethyloxindole

 $C_{10}H_{10}O_3N_2$ 

MW, 206

Cryst. from EtOH. M.p. 167°.

Brunner *et al.*, *Monatsh.*, 1931, 58, 369.

## 5-Nitro-3 : 3-dimethyloxindole.

Yellowish-red leaflets from EtOH. M.p. 262°. Sublimes at 190–220°/8 mm. Mod. sol. boiling EtOH.

*N*-Me :  $C_{11}H_{12}O_3N_2$ . MW, 220. Needles from EtOH. M.p. 203–4° (201–2°).

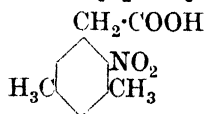
See previous reference.

## 7-Nitro-3 : 3-dimethyloxindole.

Yellow needles from EtOH. M.p. 194°. Sublimes at 145–60°/10–13 mm.

See previous reference.

## 2-Nitro-3 : 5-dimethylphenylacetic Acid

 $C_{10}H_{11}O_4N$ 

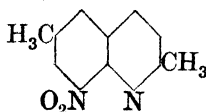
MW, 209

Needles from  $H_2O$ . M.p. 139°. Sol. EtOH,  $Et_2O$ . Mod. sol. hot  $H_2O$ .Wispek, *Ber.*, 1883, 16, 1579.

## Nitrodimethylphenylenediamine.

See Nitrodiaminoxylene.

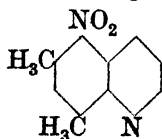
## 8-Nitro-2 : 6-dimethylquinoline (8-Nitro-6-methylquinoline)

 $C_{11}H_{10}O_2N_2$ 

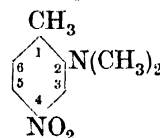
MW, 202

Orange needles from EtOH. M.p. 114°. Sol. hot EtOH. Insol.  $H_2O$ .*B*, *HCl*: grey powder.Bartow, McCollum, *J. Am. Chem. Soc.*, 1904, 26, 702.

## 5-Nitro-6 : 8-dimethylquinoline

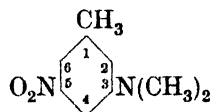
 $C_{11}H_{10}O_2N_2$ 

MW, 202

Yellowish needles from EtOH. M.p. 107–8°. Sol. usual solvents. Insol. cold  $H_2O$ . Spar. volatile in steam.Noelting, Trautmann, *Ber.*, 1890, 23, 3681.4-Nitro-*N*-dimethyl-*o*-toluidine $C_9H_{12}O_2N_2$ 

MW, 180

Yellowish-red oil. F.p. 15° (14°). B.p. 184°/77 mm., 178°/40 mm., 160°/16 mm. Sol. most org. solvents.

*B*, *HCl*: yellow plates. M.p. 197° (192°) decomp.*Methiodide*: golden-yellow needles or prisms from  $H_2O$ . M.p. 195° (rapid heat.).*Methonitrate*: yellowish needles. M.p. 230–5°.*Methochloroaurate*: yellow leaflets. M.p. 226° decomp.*Methochloroplatinate*: orange prisms. M.p. about 233° decomp.*Methopicate*: yellow needles from  $H_2O$ . M.p. about 202°.Gnehm, Blumer, *Ann.*, 1899, 304, 107.Vorländer, Siebert, *Ber.*, 1919, 52, 300.5-Nitro-*N*-dimethyl-*o*-toluidine.Leaflets or plates from EtOH. M.p. 47·5°. Very sol. EtOH,  $Et_2O$ . Mod. sol. dil.  $H_2SO_4$ .Bernthsen, *Ber.*, 1892, 25, 3133.6-Nitro-*N*-dimethyl-*o*-toluidine.Golden-yellow cryst. from  $Et_2O$ . M.p. 25–25·5°. B.p. 191–2°/100 mm. Sol. EtOH,  $Et_2O$ .v. Tatschalow, *J. prakt. Chem.*, 1902, 65, 240.5-Nitro-*N*-dimethyl-*m*-toluidine $C_9H_{12}O_2N_2$ 

MW, 180

Rhombic cryst. from  $H_2O$ . M.p. 52°. Spar. sol.  $H_2O$ . Volatile in steam.Haibach, *J. prakt. Chem.*, 1902, 65, 244.6-Nitro-*N*-dimethyl-*m*-toluidine.

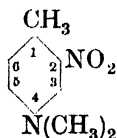
Yellow needles from EtOH. Aq. M.p. 84°.

*Methiodide*: yellow needles from  $H_2O$ . M.p. 165°.*Methonitrate*: prisms from EtOH. M.p. 195° decomp.

**Methopicate**: yellow needles or prisms from EtOH. M.p. 205°.

Vorländer, Siebert, *Ber.*, 1919, 52, 303.

### 2-Nitro-*N*-dimethyl-*p*-toluidine



$C_9H_{12}O_2N_2$

MW, 180

Orange-red prisms from EtOH. M.p. 38° (35°). Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold EtOH. Insol. H<sub>2</sub>O. Volatile in steam.

**Picrate**: yellow prisms. M.p. 147°. Spar. sol. H<sub>2</sub>O, EtOH.

**Methiodide**: yellowish prisms. M.p. 195°.

**Methobromide**: yellow prisms from EtOH. M.p. 192°.

**Methonitrate**: cryst. from EtOH. M.p. 205–20° decomp.

**Methopicate**: yellow needles. M.p. 203°.

Haibach, *J. prakt. Chem.*, 1902, 65, 248.

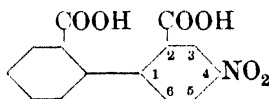
Vorländer, Siebert, *Ber.*, 1919, 52, 307.

### 3-Nitro-*N*-dimethyl-*p*-toluidine.

Cryst. from MeOH. M.p. 24.5–25°.

Pinnow, *Ber.*, 1895, 28, 3041.

### 4-Nitrodiphenic Acid (4-Nitrodiphenyl-2 : 2'-dicarboxylic acid)



$C_{14}H_8O_6N$

MW, 287

Needles from H<sub>2</sub>O. M.p. 217° (214–16°). Sol. EtOH, Et<sub>2</sub>O. Mod. sol. hot H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold H<sub>2</sub>O. Conc. H<sub>2</sub>SO<sub>4</sub> at 155° → 7-nitrofluorenone-4-carboxylic acid.

**Dichloride**: C<sub>14</sub>H<sub>7</sub>O<sub>4</sub>NCl<sub>2</sub>. MW, 324. Yellow cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 90–2°.

**Anhydride**: C<sub>14</sub>H<sub>5</sub>O<sub>5</sub>N. MW, 269. Needles from Ac<sub>2</sub>O. M.p. 207–207.5° (205–7°).

**2-(or 2')-Nitrile**: prisms from AcOH. M.p. 194–5°. **Me ester**: needles from AcOH. M.p. 123–4°.

Bell, Robinson, *J. Chem. Soc.*, 1927, 1697, 2238.

Moore, Huntress, *J. Am. Chem. Soc.*, 1927, 49, 1328.

Strasburger, *Ber.*, 1883, 16, 2347.

Schmidt, Austin, *Ber.*, 1903, 36, 3733.

Werner, Piguet, *Ber.*, 1904, 37, 4313.

### 5-Nitrodiphenic Acid (5-Nitrodiphenyl-2 : 2'-dicarboxylic acid).

Plates from H<sub>2</sub>O. M.p. 268°. Sol. MeOH, EtOH, Et<sub>2</sub>O. Mod. sol. hot H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold H<sub>2</sub>O. H<sub>2</sub>SO<sub>4</sub> at 160° → 6(?) -nitrofluorenone-4-carboxylic acid.

**Anhydride**: cryst. from Ac<sub>2</sub>O. M.p. 193–5°.

Schmidt, Austin, *Ber.*, 1903, 36, 3734.

Bell, Robinson, *J. Chem. Soc.*, 1927, 2238.

### 6-Nitrodiphenic Acid (6-Nitrodiphenyl-2 : 2'-dicarboxylic acid).

*d*-.  
[α]<sub>D</sub><sup>20</sup> + 65.2° in EtOH. Racemised by boiling Ac<sub>2</sub>O. H<sub>2</sub>SO<sub>4</sub> → 5-nitrofluorenone-4-carboxylic acid.

*l*-.  
[α]<sub>D</sub><sup>20</sup> – 66.4° in EtOH. Racemised by boiling Ac<sub>2</sub>O.

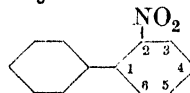
*dl*-.  
Cryst. M.p. 248–50° decomp. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, ligroin. H<sub>2</sub>SO<sub>4</sub> → 5-nitrofluorenone-4-carboxylic acid.

**Dichloride**: yellow powder. M.p. 87°.

Schmidt, Kämpf, *Ber.*, 1903, 36, 3737.

Bell, Robinson, *J. Chem. Soc.*, 1927, 1696, 2236.

### 2-Nitrodiphenyl



$C_{12}H_8O_2N$

MW, 199

Plates from EtOH. M.p. 37°. B.p. 320°, 200–5°/30 mm., 165–70°/13 mm., 160–6°/4 mm. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Sn + HCl → 2-aminodiphenyl. HNO<sub>3</sub> → 2 : 4' + 2 : 2'-dinitrodiphenyl.

Hübner, *Ann.*, 1881, 209, 341.

Bell, Kenyon, Robinson, *J. Chem. Soc.*, 1926, 1242.

Jenkins, McCullough, Booth, *Ind. Eng. Chem.*, 1930, 22, 32.

Morgan, Walls, *J. Soc. Chem. Ind.*, 1930, 49, 15r.

I.G., F.P., 764,374, (*Chem. Abstracts*, 1934, 28, 5476).

### 3-Nitrodiphenyl.

Yellow plates or needles from dil. EtOH. M.p. 62° (58.5°). Sol. EtOH, AcOH, ligroin. Volatile in steam. CrO<sub>3</sub> → *m*-nitrobenzoic

## 4-Nitrodiphenyl

acid.  $\text{Sn} + \text{HCl} \longrightarrow$  3-aminodiphenyl.  $\text{HNO}_3 \longrightarrow$  3 : 4' + 3 : 2'-dinitrodiphenyl.

Jacobson, Loeb, *Ber.*, 1903, **36**, 4083.

Fichter, Sulzberger, *Ber.*, 1904, **37**, 882.

Blakey, Scarborough, *J. Chem. Soc.*, 1927, 3003.

## 4-Nitrodiphenyl.

Yellow needles from EtOH. M.p. 114–114.5°. B.p. 223–4°/30 mm. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Mod. sol. EtOH.  $\text{CrO}_3 \longrightarrow$  *p*-nitrobenzoic acid.  $\text{Sn} + \text{HCl} \longrightarrow$  4-aminodiphenyl.  $\text{HNO}_3 \longrightarrow$  4 : 4' + 2 : 4'-dinitrodiphenyl.

Hübner, *Ann.*, 1881, **209**, 340.

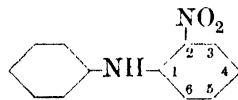
Bamberger, *Ber.*, 1895, **28**, 404.

Bell, Kenyon, Robinson, *J. Chem. Soc.*, 1926, 1242.

Jenkins, McCullough, Booth, *Ind. Eng. Chem.*, 1930, **22**, 32.

Morgan, Walls, *J. Soc. Chem. Ind.*, 1930, **49**, 15t.

## 2-Nitrodiphenylamine



$\text{C}_{12}\text{H}_{10}\text{O}_2\text{N}_2$

MW, 214

Orange plates from EtOH.Aq. M.p. 75.5°.

*N-Nitroso*: plates from MeOH. M.p. 99–100°. Hot 3% alc.  $\text{H}_2\text{SO}_4 \longrightarrow$  2-nitrodiphenylamine.

Ullmann, Nadai, *Ber.*, 1908, **41**, 1872.

Ullmann, D.R.P., 194,951, (*Chem. Zentr.*, 1908, I, 1115).

## 3-Nitrodiphenylamine.

Red plates from EtOH.Aq. M.p. 114°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin. Conc.  $\text{H}_2\text{SO}_4 + \text{HNO}_3 \longrightarrow$  violet col.

*N-Nitroso*: needles. M.p. 89–90°.

Ullmann, *Ann.*, 1907, **355**, 331.

## 4-Nitrodiphenylamine.

Yellow needles. M.p. 133°. Sol. EtOH, AcOH. Conc.  $\text{H}_2\text{SO}_4 \longrightarrow$  violet sol.  $\longrightarrow$  blue on adding  $\text{HNO}_3$ . 1% NaOH  $\longrightarrow$  intense red sol.

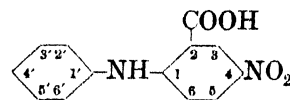
*N-Nitroso*: needles from AcOH. M.p. 133.5° (130°).

Lellmann, *Ber.*, 1882, **15**, 826.

Goldberg, D.R.P., 187,870, (*Chem. Zentr.*, 1907, II, 1465).

## 142 4-Nitrodiphenylamine-2-sulphonic Acid

### 4-Nitrodiphenylamine-2-carboxylic Acid



$\text{C}_{13}\text{H}_{10}\text{O}_4\text{N}_2$

MW, 258

Yellow needles from EtOH. M.p. 247–8°. Sublimes.

*Et ester*:  $\text{C}_{15}\text{H}_{14}\text{O}_4\text{N}_2$ . MW, 286. Yellow plates from EtOH. M.p. 121°.

*Anilide*:  $\text{C}_{19}\text{H}_{15}\text{O}_3\text{N}_3$ . MW, 333. Yellow cryst. from EtOH. M.p. 159°.

Schöpf, *Ber.*, 1890, **23**, 3441.

Grohmann, *Ber.*, 1891, **24**, 3810.

### 5 - Nitrodiphenylamine - 2 - carboxylic Acid.

Orange-yellow needles from toluene. M.p. 230°. Mod. sol. EtOH,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin.

Ullmann, Wagner, *Ann.*, 1907, **355**, 363.

### 2 - Nitrodiphenylamine - 4 - carboxylic Acid.

Red needles from EtOH. M.p. 254°. Sol. EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Mod. sol.  $\text{C}_6\text{H}_6$ . Insol. ligroin.

*Me ester*:  $\text{C}_{14}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 272. Cryst. M.p. 127°.

*Et ester*:  $\text{C}_{15}\text{H}_{14}\text{O}_4\text{N}_2$ . MW, 286. Cryst. M.p. 123°. Sol.  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol. ligroin.

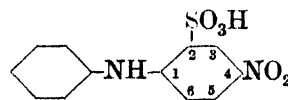
*Amide*:  $\text{C}_{13}\text{H}_{11}\text{O}_3\text{N}_3$ . MW, 257. Yellow needles from EtOH. M.p. 157°.

*Anilide*:  $\text{C}_{19}\text{H}_{15}\text{O}_3\text{N}_3$ . MW, 333. Red needles from EtOH. M.p. 215–16°. Sol. EtOH,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ ,  $\text{Me}_2\text{CO}$ , AcOH. Insol. ligroin.

Schöpf, *Ber.*, 1889, **22**, 3282; 1890, **23**, 3443.

Grohmann, *Ber.*, 1890, **23**, 3450.

### 4-Nitrodiphenylamine-2-sulphonic Acid



$\text{C}_{12}\text{H}_{10}\text{O}_5\text{N}_2\text{S}$

MW, 294

Olive-green leaflets from HCl. Very sol.  $\text{H}_2\text{O}$ , EtOH.

*Chloride*:  $\text{C}_{12}\text{H}_9\text{O}_4\text{N}_2\text{ClS}$ . MW, 312.5. Greenish-yellow needles from  $\text{Et}_2\text{O}$ . M.p. 102–4°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ .

*Amide*:  $\text{C}_{12}\text{H}_{11}\text{O}_4\text{N}_3\text{S}$ . MW, 293. Reddish-yellow cryst. from EtOH. M.p. 173°. Insol.  $\text{H}_2\text{O}$ .

*Anilide*:  $C_{18}H_{15}O_4N_3S$ . MW, 369. Greenish-yellow needles from EtOH. M.p. 164°.

Fischer, *Ber.*, 1891, **24**, 3798.

Ullmann, Dahmen, *Ber.*, 1908, **41**, 3746.

### 2-Nitrodiphenylamine-4-sulphonic Acid.

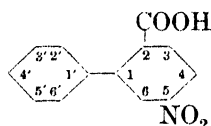
Orange cryst. from  $H_2O$ . Decomp. at 220°.

*Amide*: red cryst. from EtOH. M.p. 162°.

*Anilide*: orange-yellow needles from EtOH. M.p. 157°. Sol. EtOH,  $Me_2CO$ , AcOH.

Fischer, *Ber.*, 1891, **24**, 3791.

### 5-Nitrodiphenyl-2-carboxylic Acid



$C_{13}H_9O_4N$

MW, 243

*Nitrile*: m.p. 132-4°.

Jones, Braker, U.S.Ps., 1,922,206, 1,922,207, (*Chem. Abstracts*, 1933, **27**, 5087).

### 6-Nitrodiphenyl-2-carboxylic Acid.

Needles from 50% AcOH. M.p. 187-8°.

Sadler, Powell, *J. Am. Chem. Soc.*, 1934, **56**, 2652.

### 2'-Nitrodiphenyl-2-carboxylic Acid.

Needles from  $CHCl_3$ . M.p. 168° (165-166.5°).

*Brucine salt*: nodules from  $Me_2CO$ . M.p. 218° decomp.  $[\alpha]_D - 20.7^\circ$  in  $CHCl_3$ .

*Quinidine salt*: needles from EtOH. M.p. 196-8°.  $[\alpha]_D + 136^\circ$  in  $CHCl_3$ .

*Strychnine salt*: needles from EtOH. M.p. 216°.  $[\alpha]_D - 25^\circ$  in  $CHCl_3$ .

Bell, *J. Chem. Soc.*, 1934, 838.

See also previous reference.

### 4'-Nitrodiphenyl-2-carboxylic Acid.

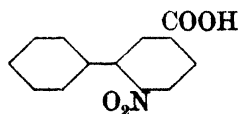
Needles from EtOH. M.p. 222-5°. Sol. hot EtOH. Prac. insol.  $H_2O$ .

Kühling, *Ber.*, 1895, **28**, 525; 1896, **29**, 166.

Grieve, Hey, *J. Chem. Soc.*, 1932, 1891 (*Footnote*).

Schmitz, *Ann.*, 1878, **193**, 123.

### 6-Nitrodiphenyl-3-carboxylic Acid



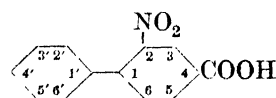
$C_{13}H_9O_4N$

MW, 243

M.p. 227°. Sublimes. (The above structure is the most probable but has not been definitely established.)

Wardner, Lowy, *J. Am. Chem. Soc.*, 1932, **54**, 2513.

### 2-Nitrodiphenyl-4-carboxylic Acid



$C_{13}H_9O_4N$

MW, 243

Pale yellow needles from EtOH. M.p. 191°.

Grieve, Hey, *J. Chem. Soc.*, 1932, 1894.

### 2'-Nitrodiphenyl-4-carboxylic Acid.

Needles from EtOH. M.p. 250°.

Grieve, Hey, *J. Chem. Soc.*, 1932, 1892; 1933, 970.

### 4'-Nitrodiphenyl-4-carboxylic Acid.

Needles from EtOH. M.p. 344-6° (340°). Prac. insol. pet. ether,  $C_6H_6$ .  $HNO_3 \rightarrow$  2:4'-dinitrodiphenyl-4-carboxylic acid.

*Et ester*:  $C_{15}H_{13}O_4N$ . MW, 271. Yellow needles from dil. EtOH. M.p. 112°.

*Chloride*:  $C_{13}H_9O_3NCl$ . MW, 261.5. M.p. 194°.

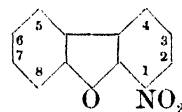
Grieve, Hey, *J. Chem. Soc.*, 1932, 1891; 1933, 971.

I.G., F.P., 735,846, (*Chem. Abstracts*, 1933, **27**, 1001); E.P., 390,556, (*Chem. Abstracts*, 1933, **27**, 4936).

### Nitrodiphenyl-2:2'-dicarboxylic Acid.

See Nitrodiphenic Acid.

### 1-Nitrodiphenylene oxide (1-Nitrodibenzofuran)



$C_{12}H_7O_3N$

MW, 213

Yellow needles from EtOH. M.p. about 110°. B.p. 190-205°/15 mm.

Borsche, Schacke, *Ber.*, 1923, **56**, 2500.

Ryan, Keane, M'Gahon, *Chem. Abstracts*, 1928, **22**, 70.

### 2-Nitrodiphenylene oxide (2-Nitrodibenzofuran).

Yellowish needles from AcOH. M.p. 181-2°. Spar. sol. hot EtOH.

Borsche, Bothe, *Ber.*, 1908, **41**, 1940.

Cullinane, *J. Chem. Soc.*, 1930, 2267.



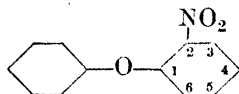
### 3-Nitrodiphenylene oxide

**3-Nitrodiphenylene oxide** (3-Nitrodibenzofuran).

Needles from EtOH. M.p. 141°.

Ryan, Keane, M'Gahon, *Chem. Abstracts*, 1928, 22, 70.

### 2-Nitrodiphenyl Ether



$C_{12}H_9O_3N$

MW, 215

Yellow liq. B.p. 235°/60 mm. part decomp., 195–7°/45 mm., 183–5°/8 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. Insol. H<sub>2</sub>O. D<sup>15</sup> 1.258, D<sup>21.5</sup> 1.2539.  $n_D^{20}$  1.575. SnCl<sub>2</sub> → 2-aminodiphenyl ether.

Hacusserrmann, Teichmann, *Ber.*, 1896, 29, 1447.

Ullmann, *ibid.*, 1880.

Jones, Cook, *J. Am. Chem. Soc.*, 1916, 38, 1537.

Lock, *Monatsh.*, 1930, 55, 177.

Brewster, Groening, *Organic Syntheses*, 1934, XIV, 67.

### 3-Nitrodiphenyl Ether.

Yellow liq. B.p. 337°/758 mm. part decomp., 202–4°/14 mm. D<sup>15</sup> 1.2451.

Ullmann, Sponagel, *Ber.*, 1905, 38, 2212; *Ann.*, 1906, 350, 103.

Lock, *Monatsh.*, 1930, 55, 179.

### 4-Nitrodiphenyl Ether.

Plates from MeOH or Et<sub>2</sub>O. M.p. 61° (56°). B.p. about 320°, 188–90°/8 mm. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH. SnCl<sub>2</sub> → 4-aminodiphenyl ether.

Hacusserrmann, Teichmann, *Ber.*, 1896, 29, 1446.

Mailhe, Murat, *Bull. soc. chim.*, 1912, 11, 446.

Brewster, Groening, *Organic Syntheses*, 1934, XIV, 66.

Rarick, Brewster, Dains, *J. Am. Chem. Soc.*, 1933, 55, 1289.

Suter, *J. Am. Chem. Soc.*, 1929, 51, 2583.

Lock, *Monatsh.*, 1930, 55, 182.

Raiford, Colbert, *J. Am. Chem. Soc.*, 1926, 48, 2659.

### Nitrodiphenyl Ether Carboxylic Acid.

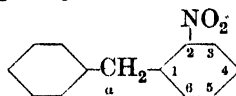
See Nitrophenoxybenzoic Acid.

### Nitrodiphenyl Ether Sulphonic Acid.

See under Nitrophenol-sulphonic Acid.

### 144 5-Nitrodiphenyl sulphide 2-carboxylic Acid

#### 2-Nitrodiphenylmethane



$C_{13}H_{11}O_2N$

MW, 213

Dark yellow liq. B.p. 183–4°/10 mm.

Carré, *Bull. soc. chim.*, 1909, 5, 119.

Gabriel, Stelzner, *Ber.*, 1896, 29, 1303.

#### 3-Nitrodiphenylmethane.

Liq. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Cannot be distilled. Non-volatile in steam.

Becker, *Ber.*, 1882, 15, 2091.

#### 4-Nitrodiphenylmethane.

Cryst. from ligroin. M.p. 31°. B.p. 202°/11 mm. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. CrO<sub>3</sub> → 4-nitrobenzophenone. Spar. volatile in steam.

Baeyer, Villiger, *Ber.*, 1904, 37, 605.

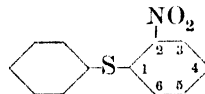
Basler, *Ber.*, 1883, 16, 2716.

$\alpha$ -Nitrodiphenylmethane (*Diphenylnitromethane*).

Oil. D<sup>0</sup> 1.1900, D<sup>20</sup> 1.1727.

Konowalow, *Ber.*, 1896, 29, 2197.

#### 2-Nitrodiphenyl sulphide



$C_{12}H_9O_2NS$

MW, 231

Yellow needles from ligroin. M.p. 80–2° (77°). Sol. EtOH, Et<sub>2</sub>O. Insol. pet. ether. Conc. H<sub>2</sub>SO<sub>4</sub> → green sol.

Bourgeois, Huber, *Bull. soc. chim.*, 1911, 9, 947.

Mauthner, *Ber.*, 1906, 39, 3597.

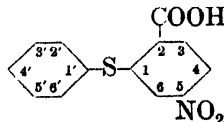
#### 4-Nitrodiphenyl sulphide.

Pale yellow prisms from ligroin. M.p. 55°. B.p. 288–2°/100 mm., 240°/25 mm.

See first reference above and also

Kehrmann, Bauer, *Ber.*, 1896, 29, 2364.

### 5-Nitrodiphenyl sulphide 2-carboxylic Acid (S-Phenyl-4-nitrothiosalicylic acid)



$C_{13}H_9O_4NS$

MW, 275

Yellow cryst. from AcOH. M.p. 210–11°. Sol. EtOH, AcOH. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin.

Mayer, *Ber.*, 1909, 42, 3066.

**2'-Nitrodiphenyl sulphide 2-carboxylic Acid** 145

**2'-Nitrodiphenyl sulphide 2-carboxylic Acid.**

Yellow cryst. from AcOH. M.p. 165-6°. Spar. sol. EtOH, CHCl<sub>3</sub>. Insol. ligroin. PCl<sub>5</sub> → 4-nitrothioxanthone.

*Me ester*: C<sub>14</sub>H<sub>11</sub>O<sub>4</sub>NS. MW, 289. Yellow cryst. from MeOH. M.p. 92°.

*Et ester*: C<sub>15</sub>H<sub>13</sub>O<sub>4</sub>NS. MW, 303. Yellow cryst. from EtOH. M.p. 75-6°.

Mayer, *Ber.*, 1909, **42**, 3060.

**3'-Nitrodiphenyl sulphide 2-carboxylic Acid.**

Yellow cryst. from AcOH. M.p. 168-9°. Sol. hot EtOH, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. SOCl<sub>2</sub> + AlCl<sub>3</sub> → 1-nitrothioxanthone.

*Me ester*: yellow cryst. M.p. 112-14°.

Mayer, *Ber.*, 1909, **42**, 3064.

**4'-Nitrodiphenyl sulphide 2-carboxylic Acid.**

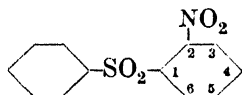
Yellow prisms from EtOH. M.p. 229-31°. Spar. sol. hot EtOH, AcOH. Insol. H<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin. Sol. alkalis → deep red sols. PCl<sub>5</sub> → 2-nitrothioxanthone.

*Me ester*: yellow cryst. from MeOH. M.p. 131-5°.

*Et ester*: yellow needles from EtOH. M.p. 127°.

Mayer, *Ber.*, 1909, **42**, 3050.

**2-Nitrodiphenyl sulphone**



C<sub>12</sub>H<sub>9</sub>O<sub>4</sub>NS

MW, 263

Cryst. from EtOH. M.p. 147-5°. Sol. warm EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH. Insol. H<sub>2</sub>O.

Ullmann, Pasdermadjian, *Ber.*, 1901, **34**, 1153.

Bourgeois, Huber, *Bull. soc. chim.*, 1911, **9**, 947.

**3-Nitrodiphenyl sulphone.**

Needles from EtOH. M.p. 80-5-81°.

Olivier, *Rec. trav. chim.*, 1915, **35**, 110.

**4-Nitrodiphenyl sulphone.**

Needles from EtOH. Aq. M.p. 143°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH. Insol. Et<sub>2</sub>O.

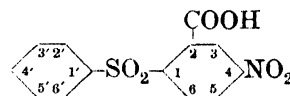
Ullmann, Pasdermadjian, *Ber.*, 1901, **34**, 1154.

Bourgeois, Huber, *Bull. soc. chim.*, 1911, **9**, 947.

Dict. of Org. Comp.—III.

**4'-Nitrodiphenyl-4-sulphonic Acid**

**5-Nitrodiphenyl sulphone 2-carboxylic Acid (4-Nitro-2-phenylsulphonebenzoic acid)**



C<sub>13</sub>H<sub>9</sub>O<sub>6</sub>NS

MW, 307

Needles from H<sub>2</sub>O. M.p. 196°. Sol. Me<sub>2</sub>CO. Spar. sol. H<sub>2</sub>O.

*Chloride*: C<sub>13</sub>H<sub>8</sub>O<sub>5</sub>NCIS. MW, 325-5. Cryst. from CHCl<sub>3</sub>. M.p. 109°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin.

*Amide*: C<sub>13</sub>H<sub>10</sub>O<sub>5</sub>N<sub>2</sub>S. MW, 306. Prisms from EtOH. M.p. 191-2°. Sol. hot EtOH, Me<sub>2</sub>CO. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, CHCl<sub>3</sub>.

Norris, *Am. Chem. J.*, 1900, **24**, 483.

**2'-Nitrodiphenyl sulphone 2-carboxylic Acid.**

Needles. M.p. 197-9°. Sol. EtOH, AcOH. Spar. sol. H<sub>2</sub>O.

*Me ester*: C<sub>14</sub>H<sub>11</sub>O<sub>6</sub>NS. MW, 321. Needles from MeOH. M.p. 127°.

Mayer, *Ber.*, 1909, **42**, 3061.

**3'-Nitrodiphenyl sulphone 2-carboxylic Acid.**

Cryst. from AcOH. M.p. 190°.

Mayer, *Ber.*, 1909, **42**, 3065.

**4'-Nitrodiphenyl sulphone 2-carboxylic Acid.**

Yellow cryst. from AcOH. M.p. 196-5°. Sol. EtOH, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>.

*Me ester*: yellow needles from MeOH. M.p. 136°.

*Et ester*: C<sub>15</sub>H<sub>13</sub>O<sub>6</sub>NS. MW, 335. Cryst. from EtOH. M.p. 101°.

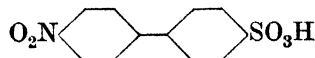
Mayer, *Ber.*, 1909, **42**, 3053.

**2-Nitrodiphenyl sulphone 4-carboxylic Acid (3-Nitro-4-phenylsulphonebenzoic acid).**

Yellowish cryst. from EtOH. M.p. 255-60°. Sol. EtOH, AcOH. Insol. C<sub>6</sub>H<sub>6</sub>.

Ullmann, Pasdermadjian, *Ber.*, 1901, **34**, 1155.

**4'-Nitrodiphenyl-4-sulphonic Acid**



C<sub>12</sub>H<sub>9</sub>O<sub>5</sub>NS

MW, 279

*Et ester*: C<sub>14</sub>H<sub>13</sub>O<sub>5</sub>NS. MW, 307. M.p. 168-9°.

*Chloride*: C<sub>12</sub>H<sub>8</sub>O<sub>4</sub>NCIS. MW, 297-5. Needles from AcOH. M.p. 178°.

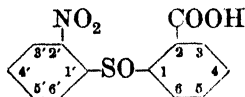
*Amide*: C<sub>12</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>S. MW, 278. M.p. 228°.

Gabriel, Dambergis, *Ber.*, 1880, **13**, 1408.

**2'-Nitrodiphenyl sulphoxide 2-carboxylic Acid**

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**2'-Nitrodiphenyl sulphoxide 2-carboxylic Acid**



$C_{13}H_9O_5NS$  MW, 291

Pale yellow leaflets from AcOH. M.p. 277°. Sol.  $PhNO_2$ . Spar. sol. EtOH. Insol.  $C_6H_6$ ,  $CHCl_3$ .

*Me ester*:  $C_{14}H_{11}O_5NS$ . MW, 305. Yellowish leaflets from MeOH. M.p. 147-8°.

*Et ester*:  $C_{15}H_{13}O_5NS$ . MW, 319. Needles from ligroin. M.p. 120°.

Mayer, *Ber.*, 1909, 42, 3060.

**3'-Nitrodiphenyl sulphoxide 2-carboxylic Acid.**

Yellow needles from AcOH. M.p. 223°.

*Me ester*: needles from AcOH. M.p. 137-8°.

See previous reference.

**4'-Nitrodiphenyl sulphoxide 2-carboxylic Acid.**

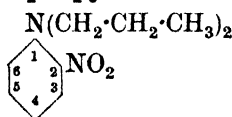
Pale yellow leaflets from AcOH or EtOH. M.p. 216-17°. Sol. EtOH. Less sol. AcOH.

*Me ester*: needles from AcOH-ligroin. M.p. 143-5°.

*Et ester*: cryst. from ligroin. M.p. 107-107.5°.

See previous reference.

**o-Nitro-N-dipropylaniline**



$C_{12}H_{18}O_2N_2$  MW, 222

Orange-yellow oil. Sol. most org. solvents. Spar. sol.  $H_2O$ .

*B, HBr*: plates. Spar. sol. EtOH.

*B, HI*: needles.

*B, HAuCl4*: yellow prisms from EtOH.

*Picrate*: golden plates from EtOH. M.p. 93-4°.

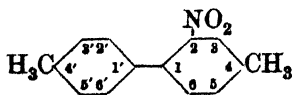
Weissenberger, *Monatsh.*, 1912, 33, 836.

**p-Nitro-N-dipropylaniline.**

Yellowish-green cryst. M.p. 59°.

Nagornow, *Chem. Zentr.*, 1898, I, 886.

**2-Nitro-4 : 4'-ditolyl (2-Nitro-4 : 4'-dimethyldiphenyl)**



$C_{14}H_{18}O_2N_2$

MW, 227

**2-Nitro-4 : 4'-ditolyl Ether**

Yellow plates from MeOH. M.p. 69-70°. B.p. 220-5°/29 mm.  $HNO_3 \rightarrow$  2 : 3'-dinitro-4 : 4'-ditolyl.

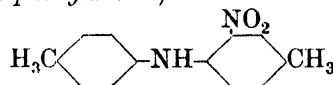
Marler, Turner, *J. Chem. Soc.*, 1932, 2393.

**3-Nitro-4 : 4'-ditolyl (3-Nitro-4 : 4'-dimethyldiphenyl).**

Plates from MeOH. M.p. 80-1°. B.p. 220-30°/20 mm.  $HNO_3 \rightarrow$  2 : 3'-dinitro-4 : 4'-ditolyl.

See previous reference.

**2-Nitro-4 : 4'-ditolylamine (2-Nitro-4 : 4'-dimethyldiphenylamine)**



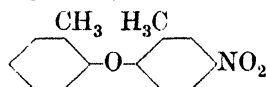
$C_{14}H_{14}O_2N_2$  MW, 242

Red leaflets from EtOH. M.p. 85°. Sol. most org. solvents.

*N-Benzoyl*: yellow prisms from EtOH. M.p. 167°. Sol. EtOH, AcOH.

Lellmann, *Ber.*, 1882, 15, 831.

**4-Nitro-2 : 2'-ditolyl Ether (4-Nitro-2 : 2'-dimethyldiphenyl ether)**

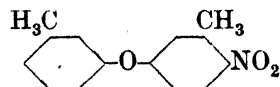


$C_{14}H_{18}O_3N$  MW, 243

Yellow needles from  $Et_2O$ . M.p. 125°. B.p. about 180°/60 mm.

Mailhe, *Bull. soc. chim.*, 1913, 13, 170.

**4-Nitro-3 : 3'-ditolyl Ether (4-Nitro-3 : 3'-dimethyldiphenyl ether)**

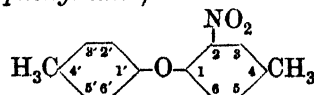


$C_{14}H_{18}O_3N$  MW, 243

M.p. 48°. B.p. 245-350°/50 mm. Sol.  $Et_2O$ .

Mailhe, *Bull. soc. chim.*, 1913, 13, 171.

**2-Nitro-4 : 4'-ditolyl Ether (2-Nitro-4 : 4'-dimethyldiphenyl ether)**



$C_{14}H_{18}O_3N$  MW, 243

Prisms from EtOH. M.p. 50°. B.p. 220°/15 mm. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. EtOH, pet. ether.

Reilly, Drumm, Barrett, *J. Chem. Soc.*, 1927, 72.

**3-Nitro-4 : 4'-ditolyl Ether** (3-Nitro-4 : 4'-dimethyldiphenyl ether).

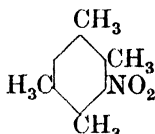
B.p. 206°/11 mm. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Less sol. EtOH. Insol. H<sub>2</sub>O.

Reilly, Barrett, *J. Chem. Soc.*, 1927, 1399.

**Nitrodulcitol.**

See under Dulcitol.

**3-Nitrodurene** (3-Nitro-1 : 2 : 4 : 5-tetramethylbenzene)



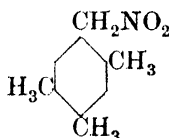
C<sub>10</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 179

Pale yellow prisms from EtOH. M.p. 112–13°.

Smith, Taylor, *J. Am. Chem. Soc.*, 1935, 57, 2463.

**ω-Nitrodurene** (1'-Nitro-1 : 2 : 4 : 5-tetramethylbenzene)



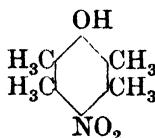
C<sub>10</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 179

Prisms from MeOH, AcOH or pet. ether. M.p. 52.5°. B.p. 143–4°/10 mm. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Sweet odour. Spar. volatile in steam.

Willstätter, Kubli, *Ber.*, 1909, 42, 4154.

**4-Nitrodurenol** (4-Nitro-2 : 3 : 5 : 6-tetramethylphenol)



C<sub>10</sub>H<sub>13</sub>O<sub>3</sub>N

MW, 195

Cryst. from EtOH. M.p. 130°. Very sol. EtOH.

Jacobsen, Schnapauff, *Ber.*, 1885, 18, 2844.

**Nitroerythritol.**

See under meso-Erythritol.

**Nitroethane.**

(i) CH<sub>3</sub>·CH<sub>2</sub>NO<sub>2</sub> (ii) CH<sub>3</sub>·CH·NO<sub>2</sub>H

C<sub>2</sub>H<sub>5</sub>O<sub>2</sub>N

MW, 75

Probably a mixture of (i) and (ii) (acinitroethane, isonitroethane). Metallic derivs. of (ii) are known.

B.p. 114–114.8°. D<sub>4</sub><sup>20</sup> 1.0685, D<sub>20</sub><sup>25</sup> 1.0461, D<sub>4</sub><sup>24.5</sup> 1.0472. Non-misc. with H<sub>2</sub>O. n<sub>D</sub><sup>20</sup> 1.40102. Heat of comb. C<sub>p</sub> (vapour) 322.3 Cal., C<sub>s</sub> 322.45 Cal. Sol. alkalis with salt formation. Fe + AcOH → ethylamine.

Na salt : white powder. Very sol. H<sub>2</sub>O. Spar. sol. EtOH.

Neogi, Chowdhuri, *J. Chem. Soc.*, 1916, 109, 701.

Kraus, D.R.P., 294,755, (*Chem. Zentr.*, 1916, II, 861).

Kissel, *Ber.*, 1882, 15, 1574.

Götting, *Ann.*, 1888, 243, 115.

Auger, *Bull. soc. chim.*, 1900, 23, 333.

Meyer, *Ann.*, 1875, 175, 88.

Krause, Swiss P., 75,523, (*Chem. Abstracts*, 1918, 12, 41).

**2-Nitroethyl Alcohol** (2-Nitroethanol, β-nitroethyl alcohol, 1-nitro-2-hydroxyethane)

NO<sub>2</sub>·CH<sub>2</sub>·CH<sub>2</sub>OH

C<sub>2</sub>H<sub>5</sub>O<sub>3</sub>N

MW, 91

Liq. with pungent odour. B.p. 194°/765 mm., 119–20°/35 mm., 102°/10 mm. D<sub>15</sub><sup>15</sup> 1.270. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Aq. sol. → no col. with FeCl<sub>3</sub>. Sn + HCl → 2-aminoethyl alcohol.

Et ether : 2-nitrodiethyl ether. C<sub>4</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 119. Liq. with sharp odour and bitter taste. B.p. 178°. D<sub>15</sub><sup>15</sup> 1.148.

Wilkendorf, Trénel, *Ber.*, 1923, 56, 619.

Wieland, Sakellarios, *Ber.*, 1919, 52, 903; 1920, 53, 201.

Demuth, Meyer, *Ann.*, 1890, 256, 29.

Henry, *Rec. trav. chim.*, 1899, 18, 259.

**N-Nitroethylamine.**

See Ethylnitramine.

**m-Nitro-N-ethylaniline**

NH·C<sub>2</sub>H<sub>5</sub>



C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

MW, 166

Reddish-yellow needles from EtOH or ligroin. M.p. 59–60°. Sol. EtOH, Et<sub>2</sub>O, ligroin. Volatile in steam.

N-Acetyl : pale yellow needles. M.p. 88–9°.

Noelting, Stricker, *Ber.*, 1886, 19, 546.

**p-Nitro-N-ethylaniline.**

Yellow cryst. with bluish-violet lustre from EtOH. M.p. 96°. Sol. warm EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. CS<sub>2</sub>, ligroin.

N-Acetyl : plates. M.p. 118–19°. Sol. EtOH,

$C_8H_9$ . Spar. sol.  $H_2O$ ,  $Et_2O$ . Insol.  $CS_2$ , ligroin.

*N*-Nitroso: straw-yellow needles. M.p. 119.5–120°.

*N*-Benzyl: yellow prisms with blue reflex from EtOH. M.p. 63°.

Schweitzer, *Ber.*, 1886, **19**, 149.

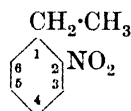
 **$\beta$ -Nitro-*N*-ethylaniline**

$C_8H_{10}O_2N_2$  MW, 166

Plates. M.p. 37°. Sol. acids and alkalis.

*N*-Nitroso: m.p. about 62°.

Wieland, Sakellarios, *Ber.*, 1919, **52**, 903.

**2-Nitroethylbenzene**

$C_8H_9O_2N$  MW, 151

F.p. — 23°. B.p. 227–8°.  $D^{24.5}$  1.126.  $n_D^{19}$  1.5407.

Schultz, Flachsländer, *J. prakt. Chem.*, 1902, **66**, 160.

Schreiner, *J. prakt. Chem.*, 1910, **81**, 558.

Cline, Reid, *J. Am. Chem. Soc.*, 1927, **49**, 3153.

**3-Nitroethylbenzene.**

B.p. 242–3°.  $D^0$  1.1345.

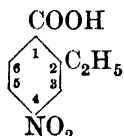
Béhal, Choay, *Bull. soc. chim.*, 1894, **11**, 211.

**4-Nitroethylbenzene.**

F.p. — 32°. B.p. 245–6°.  $D^{25}$  1.124.  $n_D^{19}$  1.5458.

Schultz, Flachsländer, *J. prakt. Chem.*, 1902, **66**, 162.

Schreiner, *J. prakt. Chem.*, 1910, **81**, 558.

**4-Nitro-2-ethylbenzoic Acid**

$C_9H_9O_4N$  MW, 195

Cryst. M.p. 126°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ , AcOH. Insol. pet. ether.  $HNO_3$  at 150°  $\longrightarrow$  4-nitrophthalic acid.

Giebe, *Ber.*, 1896, **29**, 2536.

**5-Nitro-2-ethylbenzoic Acid.**

Cryst. M.p. 164°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ , AcOH, AcOEt. Insol. pet. ether.  $HNO_3$  at 150°  $\longrightarrow$  4-nitrophthalic acid.

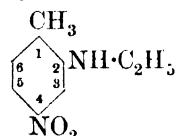
See previous reference.

**Nitroethylene**

$C_2H_3O_2N$  MW, 73

Pale yellow liq. B.p. 98.5°.  $D^{13.8}$  1.073. Sol. most org. solvents. Polymerises readily.  $SnCl_2 + HCl \longrightarrow CH_3 \cdot CHO + NH_2OH$ .  $Zn + AcOH \longrightarrow$  ethylamine. Dil.  $H_2SO_4 \longrightarrow$  2-nitroethyl alcohol.

Wieland, Sakellarios, *Ber.*, 1919, **52**, 901.

**4-Nitro-*N*-ethyl-*o*-toluidine**

$C_9H_{12}O_2N_2$  MW, 180

Yellowish-red needles from EtOH. M.p. 81–2°.

*N*-Acetyl: plates from EtOH. M.p. 90°. Very sol. EtOH. Spar. sol. pet. ether.

Ullmann, Mühlhauser, *Ber.*, 1902, **35**, 329.

Hantzsch, *Ber.*, 1910, **43**, 1673.

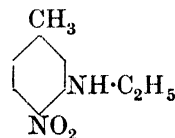
MacCallum, *J. Chem. Soc.*, 1895, **67**, 247.

**5-Nitro-*N*-ethyl-*o*-toluidine.**

Dark yellow plates from EtOH. M.p. 98°.

*N*-Acetyl: plates or prisms from EtOH.Aq. M.p. 96–7°. Very sol. EtOH.

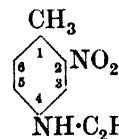
Bernthsen, *Ber.*, 1892, **25**, 3137.

**4-Nitro-*N*-ethyl-*m*-toluidine**

$C_9H_{12}O_2N_2$  MW, 180

Yellowish-red needles from EtOH.Aq. M.p. 60°.

Fischer, Rigaud, *Ber.*, 1902, **35**, 1259.

**2-Nitro-*N*-ethyl-*p*-toluidine**

$C_9H_{12}O_2N_2$  MW, 180

Red prisms or yellow needles. M.p. 50° (46–7°). Sol. EtOH, Et<sub>2</sub>O.

Noelting, Stricker, *Ber.*, 1886, **19**, 549.  
Jaubert, *Bull. soc. chim.*, 1899, **21**, 20.

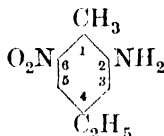
### 3-Nitro-*N*-ethyl-*p*-toluidine.

Red cryst. from EtOH. M.p. 58–9°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, hot EtOH.

*N*-Acetyl: liq. B.p. 245–50°/15 mm.

Gattermann, *Ber.*, 1885, **18**, 1483.  
Niementowski, *Ber.*, 1887, **20**, 1884.

### 6-Nitro-4-ethyl-*o*-toluidine



C<sub>9</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>

MW, 180

Yellow needles from EtOH. M.p. 96°.  
*N*-Acetyl: needles from EtOH. M.p. 166°.

Brady, Day, *J. Chem. Soc.*, 1934, 120.

### 3-Nitro-5-ethyl-*o*-toluidine.

Red prisms from pet. ether. M.p. 64°.

*N*-Acetyl: pale yellow needles from EtOH.Aq. M.p. 142°.

Morgan, Pettet, *J. Chem. Soc.*, 1934, 420.

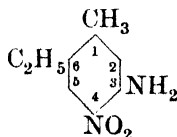
### 4-Nitro-5-ethyl-*o*-toluidine.

Golden needles. M.p. 74°.

*N*-Acetyl: m.p. 143°.

See previous reference.

### 4-Nitro-6-ethyl-*m*-toluidine



C<sub>9</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>

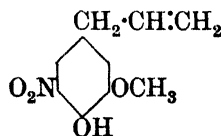
MW, 180

Orange prisms from CCl<sub>4</sub>. M.p. 90°.

*N*-Acetyl: yellow needles from 95% EtOH. M.p. 103°.

See previous reference.

**5-Nitro Eugenol** (5-Nitro-4-hydroxy-3-methoxyallylbenzene)



C<sub>10</sub>H<sub>11</sub>O<sub>4</sub>N

MW, 209

Yellow needles from EtOH. M.p. 43–4°. Volatile in steam. Sol. EtOH, Et<sub>2</sub>O. Very spar. sol. H<sub>2</sub>O.

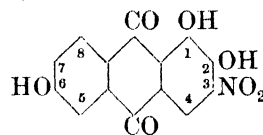
Acetyl: plates from EtOH. M.p. 61°.

Levin, Lowy, *J. Am. Chem. Soc.*, 1933, **55**, 1996.

Weselsky, Benedikt, *Monatsh.*, 1882, **3**, 388.

Klemenc, *Monatsh.*, 1912, **33**, 379.

**3-Nitroflavopurpurin** (3-Nitro-1 : 2 : 6-trihydroxyanthraquinone)



C<sub>14</sub>H<sub>7</sub>O<sub>7</sub>N

MW, 301

Red cryst.

Bayer, D.R.P., 74,562.

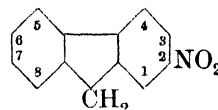
**4-Nitroflavopurpurin** (4-Nitro-1 : 2 : 6-trihydroxyanthraquinone).

Reddish-yellow cryst. from AcOH or EtOH. Decomp. above 200°. Mod. sol. EtOH, AcOH. NH<sub>3</sub>.Aq. → reddish-orange sol. NaOH → deep red sol. Conc. H<sub>2</sub>SO<sub>4</sub> → yellowish-red sol.

Bayer, D.R.P., 74,598.

M.L.B., D.R.P., 70,515.

### 2-Nitrofluorene



C<sub>13</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 211

Needles from 50% AcOH. M.p. 156° (154°).

Diels, *Ber.*, 1901, **34**, 1759.

Diels, Schill, Tolson, *Ber.*, 1902, **35**, 3289.

Kuhn, *Organic Syntheses*, 1933, XIII, 74.

### 3-Nitrofluorene.

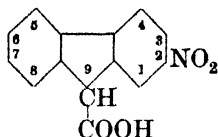
Yellow needles from CHCl<sub>3</sub>-pet. ether. M.p. 105°.

Bardout, *Chem. Zentr.*, 1932, I, 941.

### 9-Nitrofluorene.

Leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 181–2° decomp. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH. Insol. H<sub>2</sub>O, ligroin. Above m.p. → fluorenone.

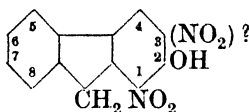
Wislicenus, Waldmüller, *Ber.*, 1908, **41**, 3338.

**2-Nitrofluorene-9-carboxylic Acid** (2-Nitrodiphenyleneacetic acid) $C_{14}H_9O_4N$ 

MW, 255

Yellow needles from  $Me_2CO-CHCl_3$ . M.p.  $186-7^\circ$ . Sol.  $Me_2CO$ , EtOH. Spar. sol.  $C_6H_6$ ,  $CHCl_3$ . Heat above m.p.  $\rightarrow$  2-nitrofluorene.

Rose, *J. Chem. Soc.*, 1932, 2361.

**1-(or 3)-Nitro-2-fluorenone** (1-(or 3)-Nitro-2-hydroxyfluorene) $C_{13}H_9O_3N$ 

MW, 227

Cryst. from MeOH. M.p.  $145-6^\circ$ .

Ruiz, *Chem. Zentr.*, 1930, II, 1074.

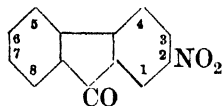
**7-Nitro-2-fluorenone** (7-Nitro-2-hydroxyfluorene).

M.p.  $219-20^\circ$  decomp.

See previous reference.

**Nitrofluorenone-carboxylic Acid.**

See Nitrohydroxyfluorene-carboxylic Acid.

**2-Nitrofluorenone** $C_{13}H_7O_3N$ 

MW, 225

Yellow cryst. M.p.  $222-3^\circ$  ( $218.5^\circ$ ). Spar. sol. cold EtOH. Sublimes easily. Conc.  $H_2SO_4 \rightarrow$  reddish-yellow sol.

*Oxime*: m.p.  $258^\circ$  (rapid heat.). *Acetyl*: pale yellow cryst. from EtOH. M.p.  $228^\circ$ .

*Hydrazone*: yellow leaflets. M.p.  $214^\circ$ .

*Phenylhydrazone*: red needles. M.p.  $210-14^\circ$  decomp. (rapid heat.). Sol.  $Me_2CO$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. EtOH, AcOH.

*Azine*: brown cryst powder from xylene. M.p.  $305-6^\circ$ .

Diels, *Ber.*, 1901, 34, 1764.

Langecker, *J. prakt. Chem.*, 1931, 132, 145.

Bardout, *Chem. Zentr.*, 1935, II, 1706.

**3-Nitrofluorenone.**

Yellowish-brown needles from EtOH. M.p.  $232^\circ$ . Spar. sol. EtOH,  $Et_2O$ .

*Oxime*: exists in two forms. (i) Yellowish-brown needles from EtOH. M.p.  $240^\circ$ . (ii) Cryst. M.p.  $217^\circ$  decomp.

Bardout, *Chem. Zentr.*, 1932, I, 941; 1935, II, 1706.

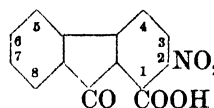
**4-Nitrofluorenone.**

Cryst. from AcOH. M.p.  $173-4^\circ$ . Sol. usual solvents. Conc.  $H_2SO_4 \rightarrow$  green sol.  $\rightarrow$  brown on warming.

*Oxime*: dark green needles. M.p.  $255-6^\circ$  decomp.

*Semicarbazone*: brown powder. Does not melt below  $350^\circ$ .

Schmidt, Bauer, *Ber.*, 1905, 38, 3742.

**2-Nitrofluorenone-1-carboxylic Acid** $C_{14}H_7O_5N$ 

MW, 269

Yellow needles. M.p.  $233-5^\circ$  decomp.

v. Braun, Manz, *Ann.*, 1932, 496, 195.

**6-Nitrofluorenone-4-carboxylic Acid.**

Cryst. from AcOH. M.p.  $282^\circ$ .

Bell, Robinson, *J. Chem. Soc.*, 1927, 2238.

**7-Nitrofluorenone-4-carboxylic Acid.**

Yellow needles from AcOH. M.p.  $262-262.5^\circ$  decomp.

*Me ester*:  $C_{15}H_9O_5N$ . MW, 283. Bright yellow needles from  $C_6H_6$ . M.p.  $199.5-200^\circ$ .

*Chloride*:  $C_{14}H_6O_4NCl$ . MW, 287.5. Yellow needles from  $C_6H_6$ . M.p.  $203.5-204^\circ$ .

*Amide*:  $C_{14}H_8O_4N_2$ . MW, 268. Yellow needles from AcOH. M.p.  $263.5-264^\circ$ . Sol. hot AcOH. Insol.  $H_2O$ , EtOH,  $CHCl_3$ ,  $Et_2O$ , chlorobenzene.

Moore, Huntress, *J. Am. Chem. Soc.*, 1927, 59, 1329.

**Nitroform.**

See Trinitromethane.

**Nitroformaldehyde** $NO_2 \cdot CHO$  $CHO_3N$ 

MW, 75

Free compound not known.

*Phenylhydrazone*: exists in two forms.  $\alpha$ -. Orange-red prisms from  $C_6H_6$ ,  $CHCl_3$  or ligroin. M.p.  $74.5-75.5^\circ$ .  $\beta$ -. Golden-yellow needles from EtOH. M.p.  $84.5-85.5^\circ$ . Less sol. than  $\alpha$ -form.

*Methylphenylhydrazone*: yellow plates or

needles. M.p. 91–2°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. Conc. H<sub>2</sub>SO<sub>4</sub> → red sol.

1-Naphthylhydrazone: dark red needles from EtOH. M.p. 120°.

Bamberger, Schmidt, Levinstein, *Ber.*, 1900, **33**, 2060.

Bamberger, Schmidt, *Ber.*, 1901, **34**, 590.

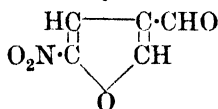
### Nitroformaldoxime.

See Methylnitrolic Acid.

### Nitroformanilide.

See under Nitroaniline.

### 5-Nitro-β-furaldehyde



C<sub>5</sub>H<sub>3</sub>O<sub>4</sub>N

MW, 141

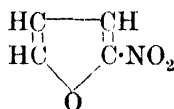
Cryst. from pet. ether. M.p. 76°.

Diacetate: cryst. from pet. ether. M.p. 87°.

Hydrazone: m.p. 122° decomp.

Gilman, Burtner, *J. Am. Chem. Soc.*, 1933, **55**, 2908.

### 2-Nitrofuran



C<sub>4</sub>H<sub>3</sub>O<sub>3</sub>N

MW, 113

Yellowish cryst. from pet. ether. M.p. 29°. Sol. H<sub>2</sub>O, Et<sub>2</sub>O. Alkalis → orange-brown sols.

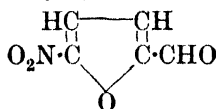
Rinkes, *Rec. trav. chim.*, 1931, **50**, 590.

Freure, Johnson, *J. Am. Chem. Soc.*, 1931, **53**, 1142.

### Nitrofuran-carboxylic Acid.

See Nitrofuroic Acid and Nitropyromucic Acid.

5-Nitrofurfural (5-Nitrofurfuraldehyde, 5-nitro-2-furoic aldehyde)



C<sub>5</sub>H<sub>3</sub>O<sub>4</sub>N

MW, 141

Cryst. from pet. ether. M.p. 35–6°. B.p. 128–32°/10 mm. Mod. sol. H<sub>2</sub>O.

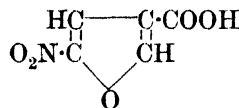
Oxime: exists in two forms. (i) Cryst. M.p. 121°. (ii) Cryst. from EtOH. M.p. 153°.

Gilman, Wright, *J. Am. Chem. Soc.*, 1930, **52**, 2552.

### 5-Nitro-α-furoic Acid.

See 5-Nitropyromucic Acid.

5-Nitro-β-furoic Acid (5-Nitrofuran-3-carboxylic acid)



C<sub>5</sub>H<sub>3</sub>O<sub>5</sub>N

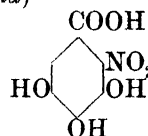
MW, 157

Cryst. from hot H<sub>2</sub>O. M.p. 138°.

Et ester: C<sub>7</sub>H<sub>7</sub>O<sub>5</sub>N. MW, 185. Cryst. from EtOH. M.p. 56°.

Gilman, Burtner, *J. Am. Chem. Soc.*, 1933, **55**, 2907.

2-Nitrogallic Acid (2-Nitro-3:4:5-trihydroxybenzoic acid)



C<sub>7</sub>H<sub>5</sub>O<sub>7</sub>N

MW, 215

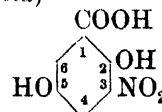
Tri-Me ether: 2-nitro-3:4:5-trimethoxybenzoic acid. C<sub>10</sub>H<sub>11</sub>O<sub>7</sub>N. MW, 257. Prisms from EtOH.Aq. M.p. 164°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. hot H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Turns yellowish-brown in air. Me ester: C<sub>11</sub>H<sub>13</sub>O<sub>7</sub>N. MW, 271. Yellowish plates or prisms from ligroin. M.p. 67–8°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Et ester: C<sub>12</sub>H<sub>15</sub>O<sub>7</sub>N. MW, 285. Yellowish cryst. from EtOH.Aq. M.p. 68–70°. Amide: C<sub>10</sub>H<sub>12</sub>O<sub>6</sub>N<sub>2</sub>. MW, 214. Needles from EtOH. M.p. 182–4°.

Tri-Et ether: 2-nitro-3:4:5-triethoxybenzoic acid. C<sub>13</sub>H<sub>17</sub>O<sub>7</sub>N. MW, 299. Needles from H<sub>2</sub>O. M.p. 104°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

Pollak, Feldscharek, *Monatsh.*, 1908, **29**, 146.

Harding, *J. Chem. Soc.*, 1911, **99**, 1592.

3-Nitrogentisic Acid (3-Nitro-2:5-dihydroxybenzoic acid)



C<sub>7</sub>H<sub>5</sub>O<sub>6</sub>N

MW, 199

Yellow cryst. + 2H<sub>2</sub>O from H<sub>2</sub>O. M.p. 230° decomp. Sol. EtOH, boiling H<sub>2</sub>O. Spar. sol. most org. solvents. Sol. NH<sub>3</sub>.Aq., carbonates, with red col. Caustic alkalis → deep violet sols. → ruby-red on dilution. FeCl<sub>3</sub> → brown col.

5-Me ether: C<sub>8</sub>H<sub>7</sub>O<sub>6</sub>N. MW, 213. Yellow needles from H<sub>2</sub>O. M.p. 181°. Very sol.



MeOH. Mod. sol. boiling  $H_2O$ .  $FeCl_3 \rightarrow$  red-dish-brown col. *Me ester*:  $C_8H_9O_6N$ . MW, 227. Yellow cryst. from MeOH. M.p.  $138-9^\circ$ . Spar. sol. cold MeOH,  $C_6H_6$ .

*Di-Me ether*: see 3-Nitro-2:5-dimethoxybenzoic Acid.

*Me ester*:  $C_8H_7O_6N$ . MW, 213. Yellow needles from  $C_6H_6$  or  $H_2O$ . M.p.  $158^\circ$ . Spar. sol. cold  $H_2O$ ,  $C_6H_6$ .  $KOH \rightarrow$  violet sol.  $\rightarrow$  orange on dilution.  $FeCl_3 \rightarrow$  green col. rapidly turning reddish-yellow.

Klemenc, *Monatsh.*, 1912, **33**, 1249.

v. Hemmelmayr, *Monatsh.*, 1913, **34**, 819.

**4-Nitrogentisic Acid** (4-Nitro-2:5-dihydroxybenzoic acid).

*5-Me ether*: yellow needles from  $C_6H_6$  or  $H_2O$ . M.p.  $191-2^\circ$ . Mod. sol.  $C_6H_6$ . *Me ester*: yellow leaflets from  $C_6H_6$ -pet. ether. M.p.  $103^\circ$ . Sol.  $C_6H_6$ .

Klemenc, *Monatsh.*, 1914, **35**, 102.

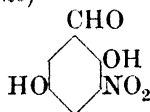
**6-Nitrogentisic Acid** (6-Nitro-2:5-dihydroxybenzoic acid).

*5-Me ether*: yellowish leaflets from boiling  $H_2O$ . M.p.  $221^\circ$  decomp. Spar. sol. hot  $C_6H_6$ . Sublimes in needles or leaflets at  $150^\circ$ . *Me ester*: cryst. from  $C_6H_6$ . M.p.  $125-6^\circ$ .

*Di-Me ether*: see 6-Nitro-2:5-dimethoxybenzoic Acid.

See previous reference.

**3-Nitrogentisic Aldehyde** (3-Nitro-2:5-dihydroxybenzaldehyde)



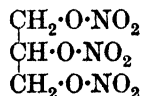
$C_7H_5O_5N$  MW, 183

*5-Me ether*:  $C_8H_7O_5N$ . MW, 197. Bright yellow needles from AcOH. M.p.  $132^\circ$ .  $FeCl_3 \rightarrow$  red col. *p-Nitrophenylhydrazone*: scarlet prisms. Decomp. at  $250^\circ$ .

*Di-Me ether*: see 3-Nitro-2:5-dimethoxybenzaldehyde.

Rubenstein, *J. Chem. Soc.*, 1925, 2000.

**Nitroglycerin** (*Glycerol trinitrate, nitroglycerol*)



$C_3H_5O_9N_3$  MW, 227

Exists in two solid forms. (i) *Labile*, cryst. M.p.  $2.0^\circ$ . (ii) *Stable*, cryst. M.p.  $13.1^\circ$ .  $D_4^{25}$

1.6144,  $D_4^{15}$  1.6009,  $D_4^{25}$  1.5918. Sol. 800 parts  $H_2O$ , 4 parts EtOH, 18 parts MeOH, 120 parts  $CS_2$ . Misc. with  $Et_2O$ ,  $CHCl_3$ , AcOH, phenol. Spar. sol. ligroin, pet. ether, glycerol. Explodes violently on rapid heating or on detonation. Possesses sweet burning taste. Used as heart stimulant, constituent of many explosives, e.g., dynamite, cordite, gelignite.

Hepworth, *J. Chem. Soc.*, 1919, **115**, 843.

Giua, *Chem. Abstracts*, 1930, **24**, 5498.

Evers, D.R.P., 513,396, (*Chem. Abstracts*, 1931, **25**, 1384).

Schmid, F.P., 707,616, (*Chem. Abstracts*, 1932, **26**, 849).

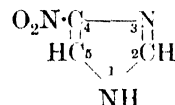
**Nitroglycerol.**

See Nitroglycerin.

**Nitroglycide.**

See under Glycide.

**4-Nitroglyoxaline** (4-Nitroiminazole)



$C_3H_3O_2N_3$

MW, 113

*N-Me*:  $C_4H_5O_2N_3$ . MW, 127. Needles from  $H_2O$ . M.p.  $133-4^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ ,  $Et_2O$ .

Hazeldine, Pyman, Winchester, *J. Chem. Soc.*, 1924, **125**, 1431.

**5-Nitroglyoxaline** (5-Nitroiminazole).

*N-Me*: prisms from  $Et_2O$ . M.p.  $55^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ . Mod. sol.  $H_2O$ ,  $Et_2O$ . *Picrate*: yellow needles from  $H_2O$ . M.p.  $153.5^\circ$ .

See previous reference.

**Nitroguaiacol.**

See under Nitrocatechol.

**Nitroguanidine**



$CH_4O_2N_4$

MW, 104

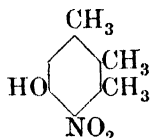
Needles or prisms from  $H_2O$ . M.p.  $232^\circ$ . decomp. Sol. 11 parts boiling  $H_2O$ , 375 parts at  $19.3^\circ$ . Spar. sol. EtOH. Insol.  $Et_2O$ . Heat of comb.  $C_p$  210.3 Cal. Decomp. by alkalis.

*B, HCl*: plates or prisms.

*B, HNO3*: plates from hot conc.  $HNO_3$ . M.p.  $140^\circ$ .

Davis, *Organic Syntheses*, Collective Vol. I, 392.

**4-Nitro-*sym.*-hemimellitenol** (2-Nitro-3:4:5-trimethylphenol, 4-nitro-5-hydroxy-1:2:3-trimethylbenzene)



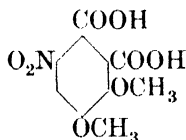
$C_9H_{11}O_3N$

MW, 181

Yellow cryst. M.p. 96–8°.

Auwers *et al.*, *Chem. Abstracts*, 1925, **19**, 2340.

**6-Nitrohemipinic Acid** (6-Nitro-3:4-dimethoxyphthalic acid)



$C_{10}H_9O_8N$

MW, 271

Yellow prisms +  $H_2O$  from  $H_2O$ . M.p. 166° (155°).  $k = 1.99 \times 10^{-2}$  at 25°. Above m.p.  $\rightarrow$  anhydride.

1-Me ester:  $C_{11}H_{11}O_8N$ . MW, 285. Cryst. from  $C_6H_6$  or  $H_2O$ . M.p. 146–7°.  $k = 1.28 \times 10^{-2}$  at 25°. Spar. sol.  $C_6H_6$ . Anilide:  $C_{17}H_{16}O_7N_2$ . MW, 360. Cryst. from  $C_6H_6$ -pet. ether. M.p. 148–9°.  $FeCl_3 \rightarrow$  red col.

2-Me ester: needles from  $C_6H_6$ . M.p. 142–4°.  $k = 1.47 \times 10^{-2}$  at 25°. Spar. sol.  $C_6H_6$ . Anilide: cryst. from  $C_6H_6$ . M.p. 170° decomp.

Di-Me ester:  $C_{12}H_{13}O_8N$ . MW, 299. Leaflets from EtOH.Aq. M.p. 83–4°.

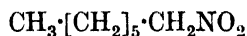
Anhydride:  $C_{10}H_7O_7N$ . MW, 253. Prisms or plates from  $C_6H_6$ . M.p. 154–5°. Sol.  $C_6H_6$ .

Wegscheider, Klemenc, *Monatsh.*, 1910, **31**, 725; 1911, **32**, 386.

Wegscheider, v. Rušnow, *Monatsh.*, 1908, **29**, 546.

Wegscheider, Strauch, *ibid.*, 568.

### 1-Nitroheptane



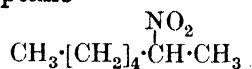
$C_7H_{15}O_2N$

MW, 145

Pale yellow oil. B.p. 193–5°.  $D^{17}_D$  0.9476. Sol. EtOH, Et<sub>2</sub>O. Insol.  $H_2O$ .  $Fe + AcOH \rightarrow n$ -heptylamine.

Worstell, *Am. Chem. J.*, 1898, **20**, 210; 1899, **21**, 223.

### 2-Nitroheptane



$C_7H_{15}O_2N$

MW, 145

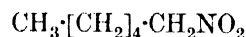
Liq. B.p. 194–8°.  $D^0$  0.9466. Sol. warm conc. KOH.

Konowalow, *J. Russ. Phys.-Chem. Soc.*, 1893, **25**, 481.

### Nitrohexahydrotoluene.

See Nitromethyleyclohexane.

### 1-Nitrohexane



$C_6H_{13}O_2N$

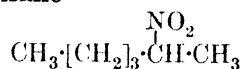
MW, 131

Stable liq. B.p. 180–1° (193–4°/765 mm.), 112°/75 mm.  $D^{20}_D$  0.9488. Sol. EtOH, aq. alkalis. Insol.  $H_2O$ . Almost odourless. Sweet taste.  $Fe + AcOH \rightarrow n$ -hexylamine.

Worstell, *Am. Chem. J.*, 1898, **20**, 207; 1899, **21**, 219.

Heury, *Rec. trav. chim.*, 1905, **24**, 358.

### 2-Nitrohexane



$C_6H_{13}O_2N$

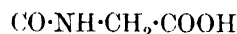
MW, 131

Liq. B.p. 176° (175–8°).  $D^0$  0.9509,  $D^{20}_D$  0.9357. Sol. boiling conc. KOH.  $Zn + AcOH$  in EtOH  $\rightarrow$  2-aminohexane + methyl butyl ketone.

Konowalow, *Compt. rend.*, 1892, **114**, 26.

Shoriugin, Lopchiev, *Ber.*, 1934, **67**, 1362.

**2-Nitrohippuric Acid** (o-Nitrobenzoyl-glycine)



$C_9H_8O_5N_2$

MW, 224

Leaflets from  $H_2O$ . M.p. 191° (188°). Sol. EtOH, hot  $H_2O$ . Spar. sol. Et<sub>2</sub>O.

Et ester:  $C_{11}H_{12}O_5N_2$ . MW, 252. Needles from EtOH. M.p. 81°.

Knoop, Oesterlin, *Z. physiol. Chem.*, 1927, **170**, 186.

**3-Nitrohippuric Acid** (m-Nitrobenzoyl-glycine).

Needles from  $H_2O$ . M.p. 165–7° (162°). Sol. EtOH, Et<sub>2</sub>O, hot  $H_2O$ .

Et ester: needles from  $H_2O$ . M.p. 75°. Sol. EtOH,  $C_6H_6$ . Spar. sol. hot  $H_2O$ .

Nitrile:  $C_9H_7O_3N_3$ . MW, 205. Leaflets from EtOH. M.p. 118°. Sol. EtOH,  $C_6H_6$ . Sol. conc. NaOH.

*Hydrazide*: needles + H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 159°.

Conrad, *J. prakt. Chem.*, 1877, **15**, 254.

Klages, Haack, *Ber.*, 1903, **36**, 1647.

Curtius, *J. prakt. Chem.*, 1914, **89**, 485.

**4-Nitrohippuric Acid** (p-Nitrobenzoyl-glycine).

Orange-red prisms from H<sub>2</sub>O. M.p. 129°. Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O.

*Et ester*: needles from EtOH. M.p. 142° (144°). Sol. Et<sub>2</sub>O, ligroin.

*Nitrile*: needles from EtOH. M.p. 145°. Sol. EtOH, AcOH. Spar. sol. Et<sub>2</sub>O, ligroin.

*Hydrazide*: yellow needles from H<sub>2</sub>O. M.p. 203.5°. *Benzylidene*: yellow powder. M.p. 216°.

*Azide*: yellow powder. M.p. 70–2° decomp. Unstable.

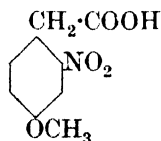
Jaffé, *Ber.*, 1874, **7**, 1673.

Sieber, Smirnow, *Monatsh.*, 1887, **8**, 90.

Klages, Haack, *Ber.*, 1903, **36**, 1648.

Curtius, *J. prakt. Chem.*, 1916, **94**, 129.

**o-Nitrohomooanisic Acid** (2-Nitro-4-methoxyphenylacetic acid)



C<sub>9</sub>H<sub>9</sub>O<sub>5</sub>N

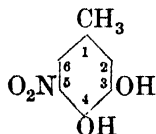
MW, 211

Yellow needles from 50% AcOH. M.p. 157–8° decomp.

Kermack, Perkin, Robinson, *J. Chem. Soc.*, 1921, **119**, 1631.

Schlittler, *Helv. Chim. Acta*, 1932, **15**, 394.

**5-Nitrohomocatechol** (5-Nitro-3 : 4-dihydroxytoluene)



C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>N

MW, 169

Golden-yellow plates from H<sub>2</sub>O. M.p. 82–3° (79–80°). Sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Volatile in steam.

*4-Me ether*: C<sub>8</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 183. *Acetyl*: yellow prisms from pet. ether. M.p. 60–1°.

*Di-Me ether*: see 5-Nitrohomoveratrol.

*3-Acetyl*: yellow needles from 80% EtOH. M.p. 104–5°.

Cousin, *Ann. chim. phys.*, 1898, **13**, 537.

Gulland, Robinson, *J. Chem. Soc.*, 1926, 1978.

**6-Nitrohomocatechol** (6-Nitro-3 : 4-dihydroxytoluene).

Yellow needles from H<sub>2</sub>O. M.p. 180–2° decomp. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. pet. ether. Insol. H<sub>2</sub>O. FeCl<sub>3</sub> → green col.

*3-Me ether*: light yellow prisms from MeOH.Aq. M.p. 138–40°. Sol. most org. solvents. Alkalis → red sols. *4-Acetyl*: yellowish needles from EtOH. M.p. 138–9°.

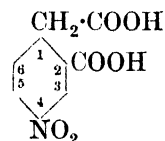
*Di-Me ether*: see 6-Nitrohomoveratrol.

Cousin, *Ann. chim. phys.*, 1898, **13**, 537.

Cardwell, Robinson, *J. Chem. Soc.*, 1915, **107**, 258.

Graesser-Thomas, Gulland, Robinson, *J. Chem. Soc.*, 1926, 1974.

**4-Nitrohomophthalic Acid**



C<sub>9</sub>H<sub>7</sub>O<sub>6</sub>N

MW, 225

Needles from H<sub>2</sub>O. M.p. 220° decomp. (215°).

*Di-Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>6</sub>N. MW, 253. Needles from MeOH. M.p. 99° (93.5°).

Borsche, Dracout, Hanau, *Ber.*, 1934, **67**, 675.

Ingold, Piggott, *J. Chem. Soc.*, 1923, **123**, 1497.

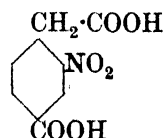
**5-Nitrohomophthalic Acid.**

Cryst. from H<sub>2</sub>O. M.p. 184.5°.

*Di-Et ester*: C<sub>13</sub>H<sub>15</sub>O<sub>6</sub>N. MW, 281. Cryst. M.p. 57°.

Heusler, Schieffer, *Ber.*, 1899, **32**, 34.

**2-Nitrohomoterephthalic Acid**



C<sub>9</sub>H<sub>7</sub>O<sub>6</sub>N

MW, 225

Leaflets from AcOH. M.p. 222°. Sol. hot H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>, pet. ether. Above m.p. → 2-nitro-*p*-toluic acid. (NH<sub>4</sub>)<sub>2</sub>S → oxindole-6-carboxylic acid.

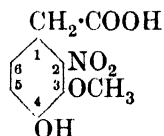
*Di-Me ester*:  $C_{11}H_{11}O_6N$ . MW, 253. Leaflets from MeOH. M.p.  $76.5^\circ$ .

*4-Nitrile*:  $C_9H_6O_4N_2$ . MW, 206. *Et ester*:  $C_{11}H_{10}O_4N_2$ . MW, 234. Yellowish needles from EtOH. M.p.  $96^\circ$ .

Borsche, Stackmann, Makaroff-Semljanski, *Ber.*, 1916, **49**, 2225.

Fileti, Cairola, *J. prakt. Chem.*, 1892, **46**, 563.

**2-Nitrohomovanillic Acid** (2-Nitro-4-hydroxy-3-methoxyphenylacetic acid)



$C_9H_6O_6N$

MW, 227

Orange-yellow needles from  $H_2O$ , leaflets from toluene. M.p.  $161^\circ$ . Sol. EtOH. NaOH  $\rightarrow$  orange sol.  $FeCl_3 \rightarrow$  faint green col.

*Benzyl ether*:  $C_{16}H_{15}O_6N$ . MW, 317. Plates from  $C_6H_6$ . M.p.  $144^\circ$ . *Benzyl ester*:  $C_{23}H_{21}O_6N$ . MW, 407. Cryst. from  $C_6H_6$ -pet. ether, m.p.  $80^\circ$ ; cryst. from EtOH, m.p.  $101^\circ$ .

*Me ether*: see 2-Nitrohomoveratric Acid.

*Carbethoxyl*: platelets +  $C_6H_6$  from  $C_6H_6$ . M.p.  $110-18^\circ$  (solvent free)  $132-3^\circ$ .

Gulland, *J. Chem. Soc.*, 1931, 2872.

**5-Nitrohomovanillic Acid** (5-Nitro-4-hydroxy-3-methoxyphenylacetic acid).

Yellow needles from  $H_2O$ . M.p.  $217^\circ$  decomp. Sol. hot  $H_2O$ , hot MeOH. Spar. sol.  $C_6H_6$ . Sol. alkalis with blood-red col.

*Me ether*: see 5-Nitrohomoveratric Acid.

*Me ester*:  $C_{10}H_{11}O_6N$ . MW, 241. Yellow needles or plates from MeOH. M.p.  $101-12^\circ$ . Very sol.  $C_6H_6$ .

Klemenc, *Monatsh.*, 1912, **33**, 382.

**6-Nitrohomovanillic Acid** (6-Nitro-4-hydroxy-3-methoxyphenylacetic acid).

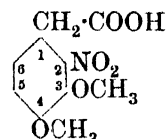
Long yellow needles from  $H_2O$ . M.p.  $184^\circ$ . NaOH  $\rightarrow$  orange-red sol.

*Me ether*: see 6-Nitrohomoveratric Acid.

*Benzyl ether*: yellow needles from EtOH. M.p.  $222^\circ$ .

Douglas, Gulland, *J. Chem. Soc.*, 1931, 2898.

**2-Nitrohomoveratric Acid** (2-Nitro-3:4-dimethoxyphenylacetic acid)



$C_{10}H_{11}O_6N$

MW, 241

Yellow leaflets from  $H_2O$ . M.p.  $146^\circ$ . Sol. most org. solvents. Mod. sol. hot  $H_2O$ .

*Nitrile*:  $C_{10}H_{10}O_4N_2$ . MW, 222. Needles from MeOH. M.p.  $68-9^\circ$ . Sol. hot  $H_2O$ .

Kay, Pictet, *J. Chem. Soc.*, 1913, **103**, 955.

Avenarius, Pschorr, *Ber.*, 1929, **62**, 323.

Slotta, Laversen, *J. prakt. Chem.*, 1934, **139**, 226.

**5-Nitrohomoveratric Acid** (5-Nitro-3:4-dimethoxyphenylacetic acid).

Yellowish needles from  $H_2O$ . M.p.  $113-14^\circ$ . Sol. boiling  $C_6H_6$ .

Klemenc, *Monatsh.*, 1912, **33**, 385.

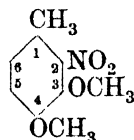
**6-Nitrohomoveratric Acid** (6-Nitro-3:4-dimethoxyphenylacetic acid).

Needles from AcOH.Aq. M.p.  $202-4^\circ$ . Sol. AcOH, AcOEt. Spar. sol.  $C_6H_6$ ,  $CHCl_3$ , cold  $H_2O$ .

*Benzyl ester*: needles from EtOH. M.p.  $117^\circ$ .

Oxford, Raper, *J. Chem. Soc.*, 1927, 419.

**2-Nitrohomoveratrol** (2-Nitro-3:4-dimethoxytoluene)



$C_9H_{11}O_4N$

MW, 197

Oil. B.p.  $115-17^\circ/1\text{ mm.}$ ,  $108-10^\circ/0.5\text{ mm.}$

Oberlin, *Arch. Pharm.*, 1925, **263**, 641.

Cf. Gulland, Robinson, *J. Chem. Soc.*, 1926, 1976.

**5-Nitrohomoveratrol** (5-Nitro-3:4-dimethoxytoluene).

Needles from EtOH. M.p.  $59^\circ$ . Very sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ . Volatile in steam.

Gulland, Robinson, *J. Chem. Soc.*, 1926, 1977.

**6-Nitrohomoveratrol** (6-Nitro-3:4-dimethoxytoluene).

Yellow needles from EtOH or ligroin. M.p.

## 1-Nitrohydantoin

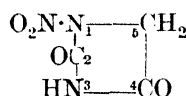
120° (118°). Spar. sol. EtOH, ligroin. Insol. H<sub>2</sub>O. Conc. H<sub>2</sub>SO<sub>4</sub> → orange-yellow sol.

Heap, Jones, Robinson, *J. Chem. Soc.*, 1927, 2022.

Cardwell, Robinson, *J. Chem. Soc.*, 1915, 107, 258.

Harding, Weizmann, *J. Chem. Soc.*, 1910, 97, 1131.

## 1-Nitrohydantoin



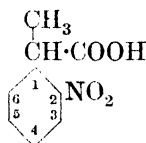
C<sub>3</sub>H<sub>3</sub>O<sub>4</sub>N<sub>3</sub> MW, 145

Needles from H<sub>2</sub>O. M.p. 170° decomp. Long heating with H<sub>2</sub>O → CO<sub>2</sub> + nitroaminoacetamide.

Franchimont, Klobbie, *Rec. trav. chim.*, 1888, 7, 12.

Franchimont, van Erp, *Rec. trav. chim.*, 1896, 15, 168.

**2-Nitrohydratropic Acid** (1-*o*-Nitrophenylpropionic acid)



C<sub>9</sub>H<sub>9</sub>O<sub>4</sub>N MW, 195

Cryst. from EtOH.Aq. M.p. 110°. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. hot H<sub>2</sub>O. Spar. sol. CS<sub>2</sub>. KMnO<sub>4</sub> → *o*-nitrobenzoic acid.

Trinius, *Ann.*, 1885, 227, 262.

**4-Nitrohydratropic Acid** (1-*p*-Nitrophenylpropionic acid).

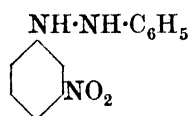
Leaflets from CS<sub>2</sub>. M.p. 87-8°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Spar. sol. cold H<sub>2</sub>O. CrO<sub>3</sub> → *p*-nitrobenzoic acid.

Nitrile: C<sub>9</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub>. MW, 176. Cryst. from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 73-5°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Opolski, Kowalski, Pilewski, *Ber.*, 1916, 49, 2282.

See also previous reference.

## *m*-Nitrohydrazobenzene



C<sub>12</sub>H<sub>11</sub>O<sub>2</sub>N<sub>3</sub> MW, 229

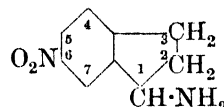
Yellow cryst. from EtOH. M.p. 85-6°.

Meisenheimer, *Ber.*, 1920, 53, 358.

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## 5-Nitrohydrindene-2 : 2-dicarboxylic Acid

**6-Nitro-1-hydrindamine** (6-Nitro-1-aminohydrindene, 6-nitro-1-indanamine)



C<sub>9</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

MW, 178

Prisms from Et<sub>2</sub>O. M.p. 60-1°.

*N*-Acetyl: needles from EtOH or AcOH.Aq. M.p. 180°.

Ingold, Piggott, *J. Chem. Soc.*, 1923, 123, 1483.

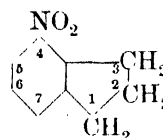
**4-Nitro-5-hydrindamine** (4-Nitro-5-aminohydrindene, 4-nitro-5-indanamine).

Orange-red cryst. M.p. 128-9°.

*N*-Benzoyl: yellow cryst. M.p. 125-6°.

Borsche, Bodenstein, *Ber.*, 1926, 59, 1909.

## 4-Nitrohydrindene (4-Nitroindane)



C<sub>9</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 163

White solid. M.p. 44-44.5°. B.p. 139°/10 mm.

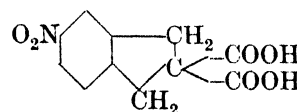
Lindner, Brukin, *Ber.*, 1927, 60, 436.

## 5-Nitrohydrindene (5-Nitroindane).

Yellow solid. M.p. 40-40.5°. B.p. 152°/14 mm.

See previous reference.

## 5-Nitrohydrindene-2 : 2-dicarboxylic Acid



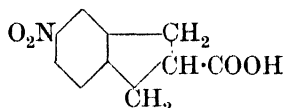
C<sub>11</sub>H<sub>9</sub>O<sub>6</sub>N

MW, 251

Pale yellow needles from H<sub>2</sub>O. M.p. 166-7°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. CHCl<sub>3</sub>. Above m.p. or heated in boiling anisole → 5-nitrohydrindenic acid. KMnO<sub>4</sub> in Na<sub>2</sub>CO<sub>3</sub>.Aq. → 4-nitrophthalic acid.

Mills, Parker, Prowse, *J. Chem. Soc.*, 1914, 105, 1538.

**5-Nitro-2-hydrindenic Acid** (5-Nitro-hydrindene-2-carboxylic acid, 5-nitroindane-2-carboxylic acid)



$C_{10}H_9O_4N$

MW, 207

*d.*

Cryst. from toluene. M.p.  $116^\circ$ .  $[\alpha]_D^{20} + 29.6^\circ$ ,  $[\alpha]_{546}^{20} + 36.4^\circ$  in toluene.

*l.*

Cryst. from  $H_2O$ . M.p.  $116^\circ$ .  $[\alpha]_D^{17} - 29.0^\circ$ ,  $[\alpha]_{546}^{17} - 36.5^\circ$  in  $H_2O$ .

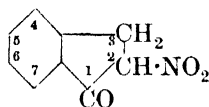
Quinine salt: m.p.  $104-7^\circ$ .  $[\alpha]_D^{20} - 102.1^\circ$ ,  $[\alpha]_{546}^{20} - 121.9^\circ$  in  $H_2O$ .

*dl.*

Needles from  $H_2O$ . M.p.  $122.5^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $Et_2O$ . Mod. sol.  $C_6H_6$ , toluene.

Mills, Parker, Prowse, *J. Chem. Soc.*, 1914, 105, 1540.

#### 2-Nitro-1-hydrindone (2-Nitroindanone-1)



$C_9H_7O_3N$

MW, 177

Yellow needles from  $C_6H_6$ -ligroin. M.p.  $117^\circ$  decomp. Sol. usual org. solvents. Mod. sol.  $H_2O$ . Spar. sol. ligroin. Reduces  $NH_3 \cdot AgNO_3$  and Fehling's.

Thiele, Weitz, *Ann.*, 1910, 377, 15.

#### 4-Nitro-1-hydrindone (4-Nitroindanone-1).

Prisms from EtOH.Aq. M.p.  $104-5^\circ$ .

Oxime: brown cryst. M.p.  $212-13^\circ$ .

Hoyer, *J. prakt. Chem.*, 1934, 139, 94.

#### 6-Nitro-1-hydrindone (6-Nitroindanone-1).

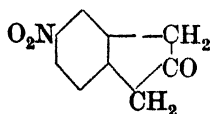
Leaflets from EtOH, AcOEt or pet. ether. M.p.  $74^\circ$ . Very sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Sol. MeOH, EtOH, AcOEt. Spar. sol. pet. ether.

Oxime: prisms from AcOEt. M.p.  $193-5^\circ$ .

Semicarbazone: decomp. at  $240^\circ$ .

Ingold, Piggott, *J. Chem. Soc.*, 1923, 123, 1485.

#### 5-Nitro-2-hydrindone (5-Nitroindanone-2)



$C_9H_7O_3N$

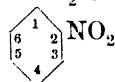
MW, 177

Brown needles with gold lustre from EtOH. M.p.  $141-141.5^\circ$ . Sol. EtOH,  $Et_2O$ , AcOH. Spar. sol.  $H_2O$ . Alkalis  $\rightarrow$  intense reddish-purple sols.

Heusler, Schieffer, *Ber.*, 1899, 32, 33.

#### *o*-Nitrohydrocinnamic Acid

$CH_2 \cdot CH_2 \cdot COOH$



$C_9H_9O_4N$

MW, 195

Yellow cryst. from  $H_2O$ . M.p.  $115^\circ$  ( $113^\circ$ ). Sn + HCl  $\rightarrow$  hydrocarbostyryl.

Me ester:  $C_{10}H_{11}O_4N$ . MW, 209. B.p.  $171-2^\circ/12$  mm.

Chloride:  $C_9H_8O_3NCl$ . MW, 213.5. Needles from pet. ether. M.p.  $43^\circ$ .

Amide:  $C_9H_{10}O_3N_2$ . MW, 194. Needles from  $H_2O$ . M.p.  $121-2^\circ$ .

Bromoamide: yellow needles. M.p.  $136-7^\circ$ .

Jaenisch, *Ber.*, 1923, 56, 2448.

#### *m*-Nitrohydrocinnamic Acid.

Yellow needles from  $H_2O$ . M.p.  $117-18^\circ$ . Sol.  $Et_2O$ , AcOH. Less sol. EtOH,  $C_6H_6$ . Spar. sol. cold  $H_2O$ ,  $CS_2$ .

Gabriel, Steudemann, *Ber.*, 1882, 15, 846.

#### *p*-Nitrohydrocinnamic Acid.

Needles from  $H_2O$ . M.p.  $163-4^\circ$ . Sol. hot EtOH. Mod. sol. hot  $Et_2O$ . Spar. sol. hot  $H_2O$ .  $CrO_3 \rightarrow$  *p*-nitrobenzoic acid.

Et ester:  $C_{11}H_{13}O_4N$ . MW, 223. Cryst. from EtOH. M.p.  $33-4^\circ$ .

Amide:  $C_9H_{10}O_3N_2$ . MW, 194. Needles from boiling  $H_2O$ . M.p.  $174-5^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol. hot  $C_6H_6$ ,  $CHCl_3$ .

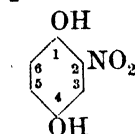
Methylamide:  $C_{10}H_{12}O_3N_2$ . MW, 208. Needles from  $H_2O$  or  $CHCl_3$ . M.p.  $166-7^\circ$ . Spar. sol. EtOH,  $Et_2O$ .

Dimethylamide:  $C_{11}H_{14}O_3N_2$ . MW, 222. M.p.  $90-1^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ .

Beilstein, Kuhlberg, *Ann.*, 1872, 163, 132.

Taverne, *Rec. trav. chim.*, 1897, 16, 255.

#### 2-Nitrohydroquinone (2-Nitroquinol)



$C_6H_5O_4N$

MW, 155

Yellow cryst. from  $H_2O$ . M.p.  $133-4^\circ$ . Very sol. EtOH,  $Et_2O$ . Sol.  $H_2O$ ,  $C_6H_6$ . Spar. sol. ligroin. Non-volatile in steam.

1-*Me ether*:  $C_7H_7O_4N$ . MW, 169. Pale yellow needles from  $H_2O$ . M.p. 97–9°. Sol. boiling  $H_2O$ ,  $C_6H_6$ . Acids  $\rightarrow$  yellow sols. Alkalis  $\rightarrow$  orange sols. *Acetyl*: needles from MeOH. M.p. 106°. Very sol. boiling MeOH. Volatile in steam.

4-*Me ether*: orange needles from EtOH or ligroin. M.p. 80° (83°). Volatile in steam.

*Di-Me ether*:  $C_8H_9O_4N$ . MW, 183. Golden-yellow needles from 50% EtOH. M.p. 72–3°. B.p. 169°/13 mm. Sublimes in needles. Sol. hot 50% EtOH.

*Et ether*:  $C_8H_9O_4N$ . MW, 183. Golden needles. M.p. 83°.

*Di-Et ether*:  $C_{10}H_{13}O_4N$ . MW, 211. Golden-yellow needles from 60% EtOH. M.p. 49°.

*Benzyl ether*:  $C_{13}H_{11}O_4N$ . MW, 245. Yellow needles from  $H_2O$ . M.p. 156–8°.

*Dibenzyl ether*:  $C_{20}H_{17}O_4N$ . MW, 335. Yellow needles from EtOH. M.p. 83° (78°).

4-*Acetyl*: yellow cryst. from EtOH. M.p. 84°.

*Diacetyl*: needles or plates from EtOH.Aq. M.p. 86° (80°).

*Dipropionyl*: pale yellow plates from EtOH.Aq. M.p. 86°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol. cold  $H_2O$ .

4-*Benzoyl*: golden-yellow needles from EtOH. M.p. 95–6°. Sol. org. solvents. Spar. sol. cold  $H_2O$ . *Acetyl*: cryst. from EtOH. M.p. 122°. Sol. hot EtOH,  $Me_2CO$ .

*Dibenzoyl*: needles from EtOH. M.p. 142°. Mod. sol. org. solvents. Insol.  $H_2O$ .

Kauffmann, Fritz, *Ber.*, 1910, **43**, 1214.

Richter, *Ber.*, 1916, **49**, 1401.

Kehrmann, Sandoz, Monnier, *Helv. Chim. Acta*, 1921, **4**, 941.

Kehrmann, Klopfenstein, *Helv. Chim. Acta*, 1923, **6**, 954.

Elbs, *J. prakt. Chem.*, 1893, **48**, 179.

Weselsky, Benedikt, *Monatsh.*, 1881, **2**, 369.

Nietzki, *Ann.*, 1882, **215**, 146.

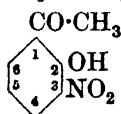
Schiff, Pellizzari, *Ann.*, 1883, **221**, 371.

Klemenc, *Monatsh.*, 1914, **35**, 91.

### Nitrohydroxyacetanilide.

See under Nitroaminophenol.

### 3-Nitro-2-hydroxyacetophenone



$C_8H_7O_4N$

MW, 181

Needles from AcOH.Aq. M.p. 89–90°. Sol.  $C_6H_6$ , AcOH. Mod. sol. boiling  $H_2O$ , EtOH.

*Oxime*: needles from  $C_6H_6$ . M.p. 182°. Turns yellow in air.

*Acetyloxime*: needles from  $C_6H_6$ . M.p. 136–7°.

Lindemann, Romanoff, *J. prakt. Chem.*, 1929, **122**, 214.

### 5-Nitro-2-hydroxyacetophenone.

Needles from AcOH.Aq. M.p. 111–12°. Sol. EtOH,  $C_6H_6$ , AcOH. Spar. sol.  $H_2O$ .

*Oxime*: needles from EtOH or  $C_6H_6$ . M.p. 231°. Sol. AcOH. Turns yellow in air.

*Acetyloxime*: cryst. from  $C_6H_6$ . M.p. 167°.

See previous reference.

### 3-Nitro-4-hydroxyacetophenone.

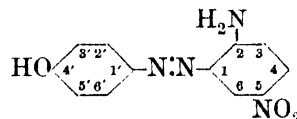
Pale yellow needles from ligroin. M.p. 135° (130°).

*Me ether*:  $C_9H_9O_4N$ . MW, 195. Needles from EtOH. M.p. 99–5°.

Stockhausen, Gattermann, *Ber.*, 1892, **25**, 3523.

Pope, *Proc. Chem. Soc.*, 1912, **23**, 332.

### 5-Nitro-4'-hydroxy-2-aminoazobenzene



$C_{12}H_{10}O_3N_4$

MW, 258

*Me ether*:  $C_{13}H_{12}O_3N_4$ . MW, 272. Brownish-yellow cryst. from AcOH. M.p. 136°.

Borsche, Exss, *Ber.*, 1923, **56**, 2353.

### 4'-Nitro-6-hydroxy-3-aminoazobenzene.

Golden cryst. from AcOH. M.p. 211°. Conc. alkalis gives indigo-blue col.

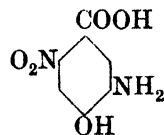
*N-Acetyl*: brown needles from AcOH. M.p. 227°. Conc.  $H_2SO_4$   $\rightarrow$  yellowish-orange col. Conc. alkali  $\rightarrow$  blue col.

Grandmougin, Freimann, *J. prakt. Chem.*, 1908, **78**, 395.

### Nitrohydroxy-*o*-aminobenzoic Acid.

See Nitrohydroxyanthranilic Acid.

### 6-Nitro-4-hydroxy-*m*-aminobenzoic Acid



$C_7H_5O_5N_2$

MW, 198

Yellow cryst. Sol. hot  $H_2O$ . Sol. alkalis with yellowish-brown col.

*Me ether*: 6-nitro-3-aminoanisic acid,  $C_8H_8O_5N_2$ . MW, 212. Pale yellow needles

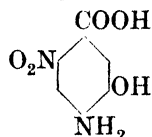
**6-Nitro-3-hydroxy-*p*-aminobenzoic Acid**

159

from  $\text{H}_2\text{O}$ . M.p.  $187-8^\circ$ . Very sol. EtOH, AcOEt,  $\text{Me}_2\text{CO}$ . Sol. hot  $\text{H}_2\text{O}$ . *N*-Acetyl: prisms from AcOH, needles from  $\text{H}_2\text{O}$ . M.p.  $259-60^\circ$ .

M.L.B., D.R.P., 184,689, (*Chem. Zentr.*, 1907, II, 764).

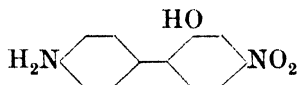
Simonsen, Rau, *J. Chem. Soc.*, 1917, 111, 235.

**6-Nitro-3-hydroxy-*p*-aminobenzoic Acid**
 $\text{C}_7\text{H}_6\text{O}_5\text{N}_2$ 

MW, 198

*Me ether*:  $\text{C}_8\text{H}_8\text{O}_5\text{N}_2$ . MW, 212. Yellow needles from  $\text{H}_2\text{O}$ . Decomp. at  $213-14^\circ$ . Sol. EtOH,  $\text{Me}_2\text{CO}$ , hot  $\text{H}_2\text{O}$ . Mod. sol. AcOEt. Spar. sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . *N*-Acetyl: leaflets from AcOH. Decomp. at  $278-80^\circ$ .

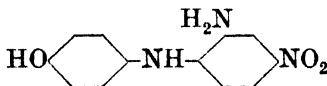
See second reference above.

**4-Nitro-2-hydroxy-4'-aminodiphenyl**
 $\text{C}_{12}\text{H}_{10}\text{O}_3\text{N}_2$ 

MW, 230

Orange-red cryst. M.p.  $145-6^\circ$ . Sol. caustic alkalis with intense red col. Sols. in dil. acids are colourless.

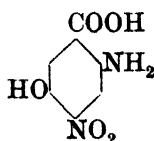
Finzi, Mangini, *Gazz. chim. ital.*, 1932, 62, 664.

**4-Nitro-4'-hydroxy-2-aminodiphenylamine**
 $\text{C}_{12}\text{H}_{11}\text{O}_3\text{N}_3$ 

MW, 245

Brownish-red needles from EtOH.Aq. M.p.  $204-5^\circ$ . Sol. conc. HCl. Mod. sol.  $\text{H}_2\text{O}$ .

Erdmann, *Ann.*, 1908, 362, 152.

**4-Nitro-5-hydroxyanthranilic Acid (4-Nitro-5-hydroxy-o-aminobenzoic acid)**
 $\text{C}_7\text{H}_6\text{O}_5\text{N}_2$ 

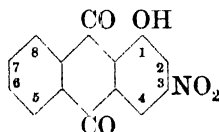
MW, 198

*Me ether*:  $\text{C}_8\text{H}_8\text{O}_5\text{N}_2$ . MW, 212. Violet-black needles from  $\text{H}_2\text{O}$ . M.p.  $217-18^\circ$ . Sol.

**5-Nitro-2-hydroxyanthraquinone**

$\text{H}_2\text{O}$  with orange-red col. *N*-Benzoyl: golden-yellow cryst. from AcOH. M.p.  $272^\circ$ . Spar. sol. EtOH, AcOH.

Ruggli, Leonhardt, *Helv. Chim. Acta*, 1924, 7, 699.

**3-Nitro-1-hydroxyanthraquinone**
 $\text{C}_{14}\text{H}_7\text{O}_5\text{N}$ 

MW, 269

Yellow leaflets from AcOH. M.p.  $247-8^\circ$  ( $240^\circ$ ). Hot NaOH  $\rightarrow$  violet sol.

Scholl, Schneider, Eberle, *Ber.*, 1904, 37, 4435.

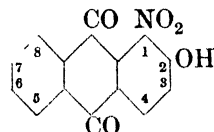
**4-Nitro-1-hydroxyanthraquinone.**

Golden-yellow needles from AcOH. M.p.  $268^\circ$ . Sol. hot AcOH, hot  $\text{PhNO}_2$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*Me ether*:  $\text{C}_{15}\text{H}_9\text{O}_5\text{N}$ . MW, 283. Cryst. Sol. EtOH, Py. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  orange-yellow sol.

Bayer, D.R.P., 205,881, (*Chem. Zentr.*, 1909, I, 881); D.R.P., 163,042, (*Chem. Zentr.*, 1905, II, 1062).

Eckert, Steiner, *Monatsh.*, 1914, 35, 1144.

**1-Nitro-2-hydroxyanthraquinone**
 $\text{C}_{14}\text{H}_7\text{O}_5\text{N}$ 

MW, 269

Cryst. M.p.  $257^\circ$ .

*Me ether*:  $\text{C}_{15}\text{H}_9\text{O}_5\text{N}$ . MW, 283. Yellowish cryst. from  $\text{PhNO}_2$ . M.p.  $271^\circ$ . Spar. sol. AcOH.

*Et ether*:  $\text{C}_{16}\text{H}_{11}\text{O}_5\text{N}$ . MW, 297. Needles from AcOH. M.p.  $243^\circ$ . Mod. sol. hot AcOH. Spar. sol. EtOH. Insol.  $\text{H}_2\text{O}$ . *Oxime*: yellow needles from AcOH. Spar. sol. AcOH.

Liebermann, Hagen, *Ber.*, 1882, 15, 1794. Benesch, *Monatsh.*, 1911, 32, 449.

**5-Nitro-2-hydroxyanthraquinone.**

Cryst. from AcOH. Caustic alkalis  $\rightarrow$  red sols.

*Me ether*: cryst. powder from MeOH, yellow leaflets from  $\text{C}_6\text{H}_6$ . M.p.  $268^\circ$ .

M.L.B., D.R.P., 167,699, (*Chem. Zentr.*, 1906, I, 1070).

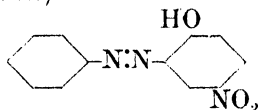


**8-Nitro-2-hydroxyanthraquinone.**

Pale yellow needles. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

*Me ether*: needles from  $\text{C}_6\text{H}_6$ . M.p.  $238^\circ$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  orange sol.

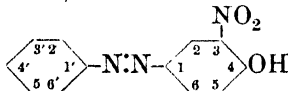
See previous reference.

**5-Nitro-2-hydroxyazobenzene** (*o*-Benzene-azo-*p*-nitrophenol)

$\text{C}_{12}\text{H}_9\text{O}_3\text{N}_3$  MW, 243

Orange-red leaflets from  $\text{AcOH.Aq.}$  M.p.  $150-1^\circ$ . Sol. usual solvents.

Auwers, Röhrig, *Ber.*, 1897, **30**, 995.

**3-Nitro-4-hydroxyazobenzene** (*p*-Benzene-azo-*o*-nitrophenol)

$\text{C}_{12}\text{H}_9\text{O}_3\text{N}_3$  MW, 243

Orange-yellow needles from ligroin. M.p.  $129^\circ$ .

*Me ether*:  $\text{C}_{13}\text{H}_{11}\text{O}_3\text{N}_3$ . MW, 257. Orange-yellow cryst. M.p.  $107^\circ$ .

*Acetyl*: yellowish-brown prisms from  $\text{AcOH.}$  M.p.  $120.5^\circ$ . Sol.  $\text{Me}_2\text{CO}$ ,  $\text{Py.}$  Less sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O.}$  Insol. pet. ether.

*Benzoyl*: yellow cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $132^\circ$ .

Borsche, Exss, *Ber.*, 1923, **56**, 2356.

Hewitt, *J. Chem. Soc.*, 1900, **77**, 99.

Valori, *Atti accad. Lincei*, 1914, **23**, II, 291.

**2'-Nitro-4-hydroxyazobenzene** (*p*-2-Nitrobenzeneazophenol).

Dark red needles from  $\text{MeOH.Aq.}$  M.p.  $162-3^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ .

*Acetyl*: yellow cryst. from  $\text{EtOH.}$  M.p.  $109^\circ$ .

Elbs, Keiper, *J. prakt. Chem.*, 1903, **67**, 581.

Elbs *et al.*, *J. prakt. Chem.*, 1924, **108**, 209.

**3'-Nitro-4-hydroxyazobenzene** (*p*-3-Nitrobenzeneazophenol).

Dark orange-red or pale yellow cryst. from toluene. M.p.  $159^\circ$  ( $147^\circ$ ). Sol.  $\text{EtOH.}$

Nölting, *Ber.*, 1887, **20**, 2998.

**4'-Nitro-4-hydroxyazobenzene** (*p*-4-Nitrobenzeneazophenol).

Reddish-brown prisms and golden-yellow

leaflets from toluene. M.p.  $212-13^\circ$ . Sol.  $\text{EtOH.}$  Insol.  $\text{H}_2\text{O.}$

*B.HCl*: dark red. M.p.  $158-167.5^\circ$ .

*Me ether*:  $\text{C}_{13}\text{H}_{11}\text{O}_3\text{N}_3$ . MW, 257. Yellowish-red needles from  $\text{EtOH.}$  M.p.  $157.5-158^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{AcOH.}$

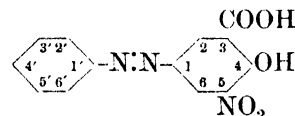
*Acetyl*: orange needles. M.p.  $147^\circ$ .

*Benzoyl*: orange needles. M.p.  $195^\circ$ .

Bamberger, *Ber.*, 1892, **25**, 846.

Nölting, *Ber.*, 1887, **20**, 2997.

Schmidt, *Ber.*, 1905, **38**, 3208.

**5-Nitro-4-hydroxyazobenzene-3-carboxylic Acid** (*5*-Benzeneazo-3-nitrosalicylic acid)

$\text{C}_{13}\text{H}_9\text{O}_5\text{N}_3$  MW, 287

Yellowish needles +  $\text{H}_2\text{O}$  from  $\text{EtOH.Aq.}$  M.p.  $197^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{AcOH}$ , boiling  $\text{H}_2\text{O.}$

*Me ester*:  $\text{C}_{14}\text{H}_{11}\text{O}_5\text{N}_3$ . MW, 301. Brown needles from  $\text{EtOH.Aq.}$  M.p.  $132-4^\circ$ .

*Et ester*:  $\text{C}_{15}\text{H}_{13}\text{O}_5\text{N}_3$ . MW, 315. Yellow needles from 95%  $\text{EtOH.}$  M.p.  $129^\circ$ .

Hewitt, Fox, *J. Chem. Soc.*, 1901, **79**, 50.

**2'-Nitro-4-hydroxyazobenzene-3-carboxylic Acid** (*5*-*o*-Nitrobenzeneazosalicylic acid).

Dark brown cryst. M.p.  $215-17^\circ$ . Sol. hot  $\text{EtOH}$ ,  $\text{AcOH.}$  Spar. sol.  $\text{H}_2\text{O.}$

Elbs, Keiper, *J. prakt. Chem.*, 1903, **67**, 583.

**3'-Nitro-4-hydroxyazobenzene-3-carboxylic Acid** (*5*-*m*-Nitrobenzeneazosalicylic acid).

Reddish-brown needles from  $\text{EtOH.}$  M.p.  $237^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ ,  $\text{AcOH.}$  Insol.  $\text{H}_2\text{O.}$  Sol. alkalis.

*Acetyl*: orange-yellow needles from  $\text{EtOH.}$  M.p.  $186^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O.}$

*Benzoyl*: yellow needles from  $\text{EtOH.}$  M.p. above  $240^\circ$ . Spar. sol.  $\text{EtOH.}$

*Me ester*:  $\text{C}_{14}\text{H}_{11}\text{O}_5\text{N}_3$ . MW, 301. Yellow needles from  $\text{EtOH.}$  M.p.  $167^\circ$ . Sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O.}$  Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  blood-red sol.

Gebek, *Ann.*, 1889, **251**, 188.

**4'-Nitro-4-hydroxyazobenzene-3-carboxylic Acid** (*5*-*p*-Nitrobenzeneazosalicylic acid).

Orange-brown needles from  $\text{AcOH.Aq.}$  M.p.  $257^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{AcOH.}$  Less sol. boiling toluene.

*Me ester*: orange needles. M.p.  $166^\circ$ . *Acetyl*: orange-yellow needles from  $\text{EtOH.}$  M.p.  $131^\circ$ .

#### 4-Nitro-4'-hydroxyazobenzene-3-carboxylic Acid 161

*Et ester* : m.p. 220-5°.

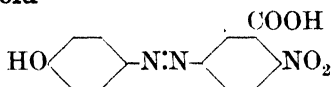
*Phenyl ester* :  $C_{19}H_{13}O_5N_3$ . MW, 363. Yellow needles from AcOH. M.p. 165°. *Acetyl* : yellow needles from EtOH. M.p. 155°.

Hewitt, Fox, *J. Chem. Soc.*, 1901, **79**, 53.

Grandmougin, Guisan, Freimann, *Ber.*, 1907, **40**, 3453.

Grandmougin, Freimann, *J. prakt. Chem.*, 1908, **78**, 396.

#### 4-Nitro-4'-hydroxyazobenzene-3-carboxylic Acid



$C_{13}H_9O_5N_3$  MW, 287

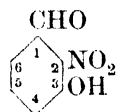
Dark red needles from AcOH. M.p. 195°. Sol. most org. solvents. Spar. sol. boiling  $H_2O$ . Blood-red sols. in alkalis.

Hewitt, Mitchell, *J. Chem. Soc.*, 1907, **91**, 1261.

#### Nitro-o-hydroxybenzaldehyde.

See Nitrosalicylaldehyde.

#### 2-Nitro-m-hydroxybenzaldehyde



$C_7H_5O_4N$  MW, 167

Needles from ligroin. M.p. 152°.

*Me ether* :  $C_8H_7O_4N$ . MW, 181. Plates from hot  $C_6H_6$ . M.p. 102°. Sol. EtOH,  $Et_2O$ , hot  $C_6H_6$ . Spar. sol.  $H_2O$ . *Oxime* : cryst. from  $H_2O$ . M.p. 170°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. hot  $H_2O$ . *p-Nitrophenylhydrazine* : orange needles. M.p. 222-3°.

*Oxime* : pale yellow needles. M.p. 172-5°.

*Phenylhydrazine* : orange-red needles. M.p. 134°.

*p-Nitrophenylhydrazine* : brick-red needles. M.p. 240-50° decomp.

Friedländer, Schenck, *Ber.*, 1914, **47**, 3043.

Rieche, *Ber.*, 1889, **22**, 2350.

Hodgson, Beard, *J. Chem. Soc.*, 1925, 875; 1927, 2375.

#### 4-Nitro-m-hydroxybenzaldehyde.

Yellow leaflets. M.p. 128°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ , ligroin.

*Me ether* : cryst. M.p. 104-5°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. ligroin. Spar. volatile in steam. *Oxime* : m.p. 148°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Insol. ligroin. *p-Nitrophenylhydrazine* : needles. M.p. 257-8°.

Dict. of Org. Comp.—III.

#### 3-Nitro-p-hydroxybenzaldehyde

*Oxime* : dark yellow needles. M.p. 164°.

*p-Nitrophenylhydrazine* : orange-red cryst. M.p. 265-6°.

See previous references.

#### 6-Nitro-m-hydroxybenzaldehyde.

Needles. M.p. 167°. Spar. sol.  $C_6H_6$ ,  $CHCl_3$ .

*Me ether* : leaflets from EtOH.Aq. M.p. 83°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ , ligroin. Volatile in steam. *Oxime* : m.p. 152°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $Me_2CO$ . Less sol.  $C_6H_6$ . *p-Nitrophenylhydrazine* : terracotta needles. M.p. 281-3°.

*Et ether* :  $C_9H_9O_4N$ . MW, 195. Cryst. M.p. 62°. *Oxime* : pale yellow cryst. M.p. 125°.

*Acetyl deriv.* : yellow needles from ligroin. M.p. 74°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin. Insol.  $H_2O$ .

*Benzoyl deriv.* : m.p. 104-5°.

*Oxime* : pale yellow needles. M.p. 179-80°.

*Semicarbazone* : pale yellow cryst. M.p. 245-60° decomp.

*Phenylhydrazine* : m.p. 185-95° decomp.

*o-Nitrophenylhydrazine* : terracotta needles. M.p. 248-50°.

*m-Nitrophenylhydrazine* : scarlet needles. M.p. 257-8°.

*p-Nitrophenylhydrazine* : deep orange-red needles. Does not melt below 300°.

Friedländer, Schenck, *Ber.*, 1914, **47**, 3043.

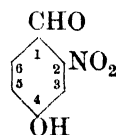
Rieche, *Ber.*, 1889, **22**, 2349.

Mason, *J. Chem. Soc.*, 1925, 1195.

Hodgson, Beard, *J. Chem. Soc.*, 1925, 875; 1927, 2375.

Pschorr, Seydel, *Ber.*, 1901, **34**, 4000.

#### 2-Nitro-p-hydroxybenzaldehyde



$C_7H_5O_4N$  MW, 167

Yellowish needles from EtOH. M.p. 67°. Sol.  $Me_2CO$ ,  $C_6H_6$ , EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ . Disagreeable odour.

*Phenylhydrazine* : red needles from EtOH. M.p. 189-90°.

Sachs, Kantorowicz, *Ber.*, 1906, **39**, 2758.

#### 3-Nitro-p-hydroxybenzaldehyde.

Yellowish needles from EtOH. M.p. 144-5° (139-40°). Sol. EtOH,  $H_2O$ . Spar. sol.  $Et_2O$ ,  $CHCl_3$ .  $FeCl_3$  → red col.

*Me ether*: see 3-Nitroanisaldehyde.

*Et ether*:  $C_9H_9O_4N$ . MW, 195. Cryst. from EtOH. M.p. 62°.

*Oxime*: prisms or needles from EtOH- $CHCl_3$ . M.p. 169°. Mod. sol. EtOH,  $Et_2O$ , AcOH. Less sol.  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. ligroin.

*Phenylhydrazone*: red needles from EtOH or AcOH. M.p. 175-6°. Sol. EtOH, AcOH,  $C_6H_6$ ,  $CHCl_3$ ,  $CS_2$ . Less sol.  $Et_2O$ . *O-Acetyl*: orange-red needles from EtOH. M.p. 134-5°. Sol.  $Et_2O$ ,  $C_6H_6$ , AcOH,  $CHCl_3$ . Mod. sol. EtOH. *N-Acetyl*: golden-yellow needles from EtOH. M.p. 193-4°. Sol.  $C_6H_6$ ,  $CHCl_3$ , AcOH. Mod. sol. EtOH.

*p-Nitrophenylhydrazone*: orange needles from AcOH. M.p. 247-9° decomp.

*p-Bromophenylhydrazone*: red needles from AcOH. M.p. 192-3°.

Paal, *Ber.*, 1895, **28**, 2413.

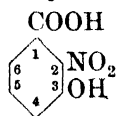
Auwers, Rohrig, *Ber.*, 1897, **30**, 996.

Fishman, *J. Am. Chem. Soc.*, 1920, **42**, 2299.

### Nitro-*o*-hydroxybenzoic Acid.

See Nitrosalicylic Acid.

### 2-Nitro-*m*-hydroxybenzoic Acid



$C_7H_5O_5N$

MW, 183

Plates and prisms +  $H_2O$  from  $H_2O$ . M.p. 178°. Sol. EtOH,  $Et_2O$ . Mod. sol.  $H_2O$ . Very sweet.

*Me ether*: 2-nitro-*m*-methoxybenzoic acid.  $C_8H_7O_5N$ . MW, 197. Leaflets from  $H_2O$ . M.p. 251° decomp. Sol. EtOH,  $Me_2CO$ , AcOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .  $NH_3$  at 180° → 2-nitro-3-aminobenzoic acid.

*Et ester*:  $C_9H_9O_5N$ . MW, 211. Cryst. from EtOH. M.p. 124°. Very sol. EtOH. *Et ether*:  $C_{11}H_{13}O_5N$ . MW, 239. Plates from EtOH. M.p. 53-4°.  $NH_3$ . Aq. at 150° → 2-nitro-3-aminobenzoic acid.

Griess, *Ber.*, 1887, **20**, 407.

Rieche, *Ber.*, 1889, **22**, 2352.

Thieme, *J. prakt. Chem.*, 1891, **43**, 467.

### 4-Nitro-*m*-hydroxybenzoic Acid.

Yellow leaflets from  $H_2O$ . M.p. 230°. Very spar. sol.  $H_2O$ .

*Me ether*: 4-nitro-*m*-methoxybenzoic acid.  $C_8H_7O_5N$ . MW, 197. Needles. M.p. 233°.

*Et ether*:  $C_9H_9O_5N$ . MW, 211. Pale yellow needles from EtOH. M.p. 216-5°. Sol. EtOH,

$Et_2O$ . Spar. sol.  $H_2O$ . *Et ester*:  $C_{11}H_{13}O_5N$ . MW, 239. Needles. M.p. 60-1°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH. Insol.  $H_2O$ . *Amide*:  $C_9H_{10}O_4N_2$ . MW, 210. Golden-yellow needles from  $H_2O$ . M.p. 202°. Sol. EtOH, AcOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ .

*Me ester*:  $C_8H_7O_5N$ . MW, 197. Needles from EtOH. M.p. 92°.

*Et ester*:  $C_9H_9O_5N$ . MW, 211. Yellowish prisms from EtOH. M.p. 84°. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

Brenans, Prost, *Compt. rend.*, 1924, **178**, 1285.

Einhorn, Pfyl, *Ann.*, 1900, **311**, 44.

See also previous references.

### 5-Nitro-*m*-hydroxybenzoic Acid.

Leaflets or plates. M.p. 167°. Sol. EtOH,  $Et_2O$ , hot  $H_2O$ .

*Anilide*: needles from AcOH.Aq. M.p. 232°. Sol. AcOH. Mod. sol. EtOH. Insol.  $H_2O$ ,  $Et_2O$ .

*Azide*: reddish-yellow ppt. Decomp. on heating. Spar. sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

Curtius, Riedel, *J. prakt. Chem.*, 1907, **76**, 260.

Griess, *Ber.*, 1887, **20**, 407.

### 6-Nitro-*m*-hydroxybenzoic Acid.

Yellow needles or prisms from  $H_2O$ . M.p. 171.5-172°. Very sol.  $H_2O$ , EtOH,  $Et_2O$ .

*Me ether*: 6-nitro-*m*-methoxybenzoic acid. M.p. 132-3°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , AcOH. Insol. ligroin.

Rieche, *Ber.*, 1889, **22**, 2354.

Beyer, *Rec. trav. chim.*, 1921, **40**, 624.

Brenans, *Compt. rend.*, 1924, **178**, 1285.

See also last reference above.

### 2-Nitro-*p*-hydroxybenzoic Acid.

*Me ether*: see 2-Nitroanisic Acid.

### 3-Nitro-*p*-hydroxybenzoic Acid.

Needles or leaflets from  $H_2O$ . M.p. 186-7° (182-3°). Sol. EtOH,  $Et_2O$ , hot  $H_2O$ . No col. with  $FeCl_3$ .

*Me ether*: see 3-Nitroanisic Acid.

*Et ether*:  $C_9H_9O_5N$ . MW, 211. Cryst. M.p. 200-1°. *Et ester*:  $C_{11}H_{13}O_5N$ . MW, 239. Cryst. from EtOH. M.p. 64°. *Chloride*:  $C_9H_8O_5NCl$ . MW, 229.5. M.p. 81-2°. B.p. 215-16°/20 mm.

*Phenyl ether*: see 3-Nitro-*p*-phenoxybenzoic Acid.

*Me ester*:  $C_8H_7O_5N$ . MW, 197. Yellow needles from EtOH.Aq. M.p. 75-6° (70-1°). *Propyl ether*:  $C_{11}H_{13}O_5N$ . MW, 239. Oil. B.p. 144-6°/11 mm. *Benzoyl*: needles from EtOH. M.p. 95°. *p-Toluenesulphonyl*: m.p. 86°.

*Et ester*:  $C_9H_9O_5N$ . MW, 211. Yellowish-

red prisms from EtOH. M.p. 75–6° (69°). Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*Propyl ester*: C<sub>10</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 225. Yellow cryst. M.p. 60–1°.

*Butyl ester*: C<sub>11</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 239. Yellow oil. B.p. 174–6°/8 mm.

*Isoamyl ester*: C<sub>12</sub>H<sub>15</sub>O<sub>5</sub>N. MW, 253. Yellow needles from EtOH. M.p. 59°. B.p. 117–19°/2 mm.

*Benzyl ester*: C<sub>14</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 273. Yellowish-brown needles from EtOH. M.p. 82°.

*Nitrile*: 2-nitro-4-cyanophenol. C<sub>7</sub>H<sub>4</sub>O<sub>3</sub>N<sub>2</sub>. MW, 164. Yellowish leaflets from very dil. EtOH. M.p. 144–5°. *Acetyl*: m.p. 113–14°.

*Carbathoxyl*: needles from EtOH.Aq. M.p. 176–7°.

Griess, *Ber.*, 1887, **20**, 408.

Diepolder, *Ber.*, 1896, **29**, 1756.

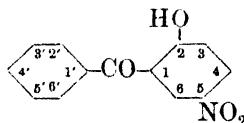
Thieme, *J. prakt. Chem.*, 1891, **43**, 453.

Einhorn, *Pfyl. Ann.*, 1900, **311**, 67.

Biehringer, *Borsum, Ber.*, 1915, **48**, 1316.

Sabalitschka, Tiedge, *Arch. Pharm.*, 1934, **272**, 383.

### 5-Nitro-2-hydroxybenzophenone



C<sub>13</sub>H<sub>9</sub>O<sub>4</sub>N MW, 243

Cryst. from EtOH. M.p. 124–124.5°.

Ullmann, Mallet, *Ber.*, 1898, **31**, 1696.

### 4'-Nitro-2-hydroxybenzophenone.

Yellow prisms. M.p. 111–13°. Sol. hot EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin.

*Me ether*: C<sub>14</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 257. Prisms and leaflets from EtOH or ligroin. M.p. 117–19°. Sol. hot EtOH, AcOH. Spar. sol. Et<sub>2</sub>O, hot ligroin.

Auwers, *Ber.*, 1903, **36**, 3896.

### 3-Nitro-4-hydroxybenzophenone.

Yellowish-brown rhombic cryst. from MeOH. M.p. 120–1°.

*Me ether*: C<sub>14</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 257. Yellow cryst. from MeOH. M.p. 105°. *Oxime*: yellow. M.p. 179°.

Borsche, *Ber.*, 1917, **50**, 1354.

v. Alphen, *Rec. trav. chim.*, 1930, **49**, 384.

### 2'-Nitro-4-hydroxybenzophenone.

*Et ether*: C<sub>15</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 271. Yellow prisms from EtOH. M.p. 115°. Sol. most org. solvents except cold EtOH, ligroin.

Auwers, *Ber.*, 1903, **36**, 3891.

### 3'-Nitro-4-hydroxybenzophenone.

Yellowish prisms from EtOH.Aq. M.p. 173°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, CS<sub>2</sub>. Spar. sol. ligroin.

*Me ether*: C<sub>14</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 257. Pale yellow cryst. from MeOH. M.p. 95°.

*Et ether*: C<sub>15</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 271. Prisms from EtOH–ligroin. M.p. 79–81°. Sol. most org. solvents.

*Phenyl ether*: C<sub>19</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 319. Cryst. M.p. 87–8°. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

v. Alphen, *Rec. trav. chim.*, 1930, **49**, 389.

See also previous reference.

### 4'-Nitro-4-hydroxybenzophenone.

Yellow needles. M.p. 190–2°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. ligroin, pet. ether.

*Me ether*: needles from EtOH or AcOH. M.p. 121°. Spar. sol. EtOH, Et<sub>2</sub>O, AcOH.

*Et ether*: yellowish needles. M.p. 112°.

*Phenyl ether*: m.p. 121–2°. Conc. H<sub>2</sub>SO<sub>4</sub> → brownish-red sol.

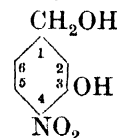
*Acetyl*: needles from EtOH. M.p. 131°. Spar. sol. EtOH, AcOH. Almost insol. Et<sub>2</sub>O, ligroin, pet. ether.

Auwers, *Ber.*, 1903, **36**, 3893.

### Nitro-*o*-hydroxybenzyl Alcohol.

See Nitrosaligenin.

### 4-Nitro-*m*-hydroxybenzyl Alcohol



C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>N MW, 169

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 97°. Spar. sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.

Lock, *Ber.*, 1929, **62**, 1185.

### 6-Nitro-*m*-hydroxybenzyl Alcohol.

Cryst. from H<sub>2</sub>O. M.p. 120.5°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Mod. sol. hot H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, toluene. Alkalis → intense yellow sols. FeCl<sub>3</sub> → reddish-violet col.

Lock, *Ber.*, 1929, **62**, 1184.

### 3-Nitro-*p*-hydroxybenzyl Alcohol.

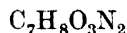
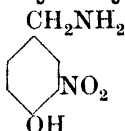
Prisms from H<sub>2</sub>O. M.p. 97°. Sol. Et<sub>2</sub>O, AcOH. Spar. sol. EtOH, C<sub>6</sub>H<sub>6</sub>.

*4-Me ether*: 3-nitroanisyl alcohol. C<sub>8</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 183. Needles from H<sub>2</sub>O. M.p. 69°.

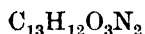
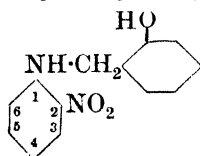
*4-Et ether*: C<sub>9</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 197. Needles from H<sub>2</sub>O. M.p. 66°.

Fishman, *J. Am. Chem. Soc.*, 1920, **42**, 2292.

Hart, Hirschfelder, *ibid.*, 2683.

**Nitro-*o*-hydroxybenzylamine.***See* Nitrosalicylamine.**3-Nitro-*p*-hydroxybenzylamine**

MW, 168

Orange-red needles + H<sub>2</sub>O from H<sub>2</sub>O. At 115° loses H<sub>2</sub>O → yellow solid, m.p. 225°.*B, HCl*: m.p. 242° decomp.*N-Benzoyl*: yellow needles from EtOH. M.p. 137°.Einhorn *et al.*, *Ann.*, 1905, **343**, 243.**2-Nitro-*N*-*o*-hydroxybenzylaniline**

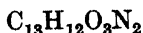
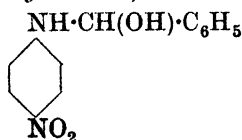
MW, 244

Dark red plates or needles. M.p. 125°. Very sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Mod. sol. EtOH.*O-Acetyl*: yellow needles from EtOH.Aq. M.p. 93°. Sol. Et<sub>2</sub>O. Insol. ligroin.Paal, Härtel, *Ber.*, 1899, **32**, 2059.**3-Nitro-*N*-*o*-hydroxybenzylaniline.**

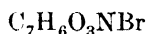
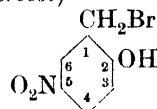
Orange needles from EtOH. M.p. 115°.

*N-Acetyl*: prisms from EtOH.Aq. M.p. 126°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.*O*: *N-Diacetyl*: needles from EtOH.Aq. M.p. 99°. Sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, warm EtOH.*See* previous reference.**4-Nitro-*N*-*o*-hydroxybenzylaniline.**

Golden-yellow plates from EtOH.Aq. M.p. 138°. Sol. org. solvents except ligroin.

*O*: *N-Diacetyl*: needles from EtOH. M.p. 79°. Sol. C<sub>6</sub>H<sub>6</sub>, hot CHCl<sub>3</sub>, AcOH.*See* previous reference.**4-Nitro-*N*-*α*-hydroxybenzylaniline** (*ω*-*p*-Nitroanilinobenzyl alcohol)

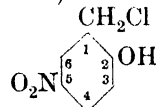
MW, 244

Yellow prisms. M.p. 85–6°. In vacuo over H<sub>2</sub>SO<sub>4</sub> → benzaldehyde-4-nitroanil.*B, HCl*: yellow cryst. powder. M.p. about188°. Sol. EtOH. Insol. Et<sub>2</sub>O. Decomp. by cold H<sub>2</sub>O.Dimroth, Zoeppritz, *Ber.*, 1902, **35**, 989.  
v. Miller, Plöchl, Rohde, *Ber.*, 1892, **25**, 2054.**5-Nitro-*o*-hydroxybenzyl bromide** (*α*-Bromo-5-nitro-*o*-cresol)

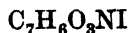
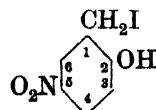
MW, 232

Leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 147°. Sol. EtOH. Mod. sol. C<sub>6</sub>H<sub>6</sub>, AcOH. Spar. sol. ligroin.Auwers, *Ber.*, 1906, **39**, 3173.**3-Nitro-*p*-hydroxybenzyl bromide** (*α*-Bromo-3-nitro-*p*-cresol).

Yellow needles from AcOH or pet. ether. M.p. 83–5° (82°). Sol. most solvents. Spar. sol. AcOH, pet. ether, ligroin.

*Me ether*: C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>NBr. MW, 246. Pale yellow needles from pet. ether. M.p. 108°.Bayer, D.R.P., 132,475, (*Chem. Zentr.*, 1902, II, 81).Shoesmith, Hetherington, Slater, *J. Chem. Soc.*, 1924, **125**, 1316.**5-Nitro-*o*-hydroxybenzyl chloride** (*α*-Chloro-5-nitro-*o*-cresol)

MW, 187.5

Needles or plates from C<sub>6</sub>H<sub>6</sub>. M.p. 132° (128°). Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O, ligroin. Hot H<sub>2</sub>O → 5-nitrosaligenin.Einhorn, *Ann.*, 1905, **343**, 245.Bayer, D.R.P., 132,475, (*Chem. Zentr.*, 1902, II, 81).**3-Nitro-*p*-hydroxybenzyl chloride** (*α*-Chloro-3-nitro-*p*-cresol).Yellow needles from C<sub>6</sub>H<sub>6</sub>, ligroin or EtOH, leaflets from pet. ether. M.p. 75° (72°).*See* last reference above and alsoStoermer, Behn, *Ber.*, 1901, **34**, 2459.**5-Nitro-*o*-hydroxybenzyl iodide** (*α*-Iodo-5-nitro-*o*-cresol)

MW, 279

### 3-Nitro-*p*-hydroxybenzyl iodide

Cryst. M.p. 169°.

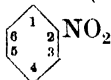
Bayer, D.R.P., 132,475, (*Chem. Zentr.*, 1902, II, 81).

### 3-Nitro-*p*-hydroxybenzyl iodide ( $\alpha$ -Iodo-3-nitro-*p*-cresol).

Pale yellow cryst. M.p. 112°.

See previous reference.

### 2-Nitro- $\alpha$ -hydroxycinnamic Acid (2-Nitro-phenylpyruvic acid, enol form)



$\text{C}_9\text{H}_7\text{O}_5\text{N}$

MW, 209

*Me ether*:  $\text{C}_{10}\text{H}_9\text{O}_5\text{N}$ . MW, 223. Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 160–1°. Gives no col. with  $\text{FeCl}_3$ . *Me ester*:  $\text{C}_{11}\text{H}_{11}\text{O}_5\text{N}$ . MW, 237. Needles from ligroin. M.p. 67.5–68°. Spar. sol. EtOH. *Et ester*:  $\text{C}_{12}\text{H}_{13}\text{O}_5\text{N}$ . MW, 251. Cryst. from EtOH. M.p. 46–7°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ .

*Et ether*:  $\text{C}_{11}\text{H}_{11}\text{O}_5\text{N}$ . MW, 237. Needles from  $\text{C}_6\text{H}_6$ . M.p. 146–7°. *Me ester*:  $\text{C}_{12}\text{H}_{13}\text{O}_5\text{N}$ . MW, 251. Brown needles from ligroin. M.p. 49–50°.

*Me ester*:  $\text{C}_{10}\text{H}_9\text{O}_5\text{N}$ . MW, 223. Yellow cryst. from EtOH. M.p. 92–93.5°.

*Et ester*:  $\text{C}_{11}\text{H}_{11}\text{O}_5\text{N}$ . MW, 237. Cryst. from ligroin. M.p. 70–1°.  $\text{FeCl}_3 \rightarrow$  dark green col. Cu acetate  $\rightarrow$  green col. *Acetyl*: prisms from  $\text{C}_6\text{H}_6$ . M.p. 95–6°. *Benzoyl*: plates from EtOH or ligroin. M.p. 46–7°. Does not react with  $\text{FeCl}_3$ . Does not decolourise  $\text{KMnO}_4$  in  $\text{Me}_2\text{CO}$ .

Wislicenus, Thoma, *Ann.*, 1924, 436, 51.

### 4-Nitro- $\alpha$ -hydroxycinnamic Acid (4-Nitro-phenylpyruvic acid, enol form).

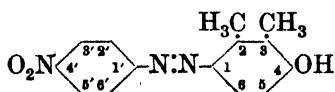
*Et ester*: yellow prisms. M.p. 106°. Mod. sol. usual solvents. Insol. ligroin. Alkalis  $\rightarrow$  deep red sols.  $\text{FeCl}_3$  in dil. EtOH  $\rightarrow$  brownish-green col.

Wislicenus, Schultz, *Ann.*, 1924, 436, 58.

### $\beta$ -Nitro- $\alpha$ -hydroxydibenzyl.

See  $\beta$ -Nitro- $\alpha$ -hydroxy-*sym*-diphenylethane.

### 4'-Nitro-4-hydroxy-2 : 3-dimethylazobenzene



$\text{C}_{14}\text{H}_{13}\text{O}_3\text{N}_3$

MW, 271

*Me ether*:  $\text{C}_{15}\text{H}_{15}\text{O}_3\text{N}_3$ . MW, 285. Dark red needles from EtOH. M.p. 142°. Sol.  $\text{C}_6\text{H}_6$ ,

### 165 3-Nitro-4-hydroxy-2 : 5-dimethylbenzaldehyde

AcOH. Spar. sol. EtOH, MeOH. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  bluish-red sol.

Auwers, Michaelis, *Ber.*, 1914, 47, 1294.

### 4'-Nitro-4-hydroxy-2 : 5-dimethylazobenzene.

Violet needles from AcOH. M.p. 222–3°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, AcOH, ligroin.

*Me ether*: bluish-red needles from AcOH. M.p. 163–4°. Sol.  $\text{C}_6\text{H}_6$ . Mod. sol. EtOH, AcOH. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  red sol.

Auwers, Rietz, *Ann.*, 1907, 356, 164 (Note).

### 4'-Nitro-4-hydroxy-2 : 6-dimethylazobenzene.

Red needles from AcOH. M.p. 166–7°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Mod. sol. AcOH. Spar. sol. EtOH, ligroin.

*Me ether*: violet needles from AcOH. M.p. 119–20°. Sol.  $\text{C}_6\text{H}_6$ . Mod. sol. AcOH. Spar. sol. EtOH, pet. ether.

*Isopropyl ether*:  $\text{C}_{17}\text{H}_{19}\text{O}_3\text{N}_3$ . MW, 313. Brownish-violet needles from ligroin. M.p. 92–3°. Sol. usual solvents. Spar. sol. EtOH, pet. ether.

*Allyl ether*:  $\text{C}_{17}\text{H}_{17}\text{O}_3\text{N}_3$ . MW, 311. Brownish-red needles from pet. ether. M.p. 102–3°.

Auwers, Rietz, *Ann.*, 1907, 356, 165 (Note).

Auwers, Michaelis, *Ber.*, 1914, 47, 1293.

Ler, *Chem. Abstracts*, 1934, 28, 4715.

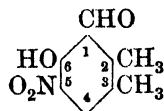
### 4'-Nitro-2-hydroxy-3 : 5-dimethylazobenzene.

Red needles from AcOH or EtOH. M.p. 194–5° (193°). Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, AcOH, ligroin.

See last reference above and also

Auwers, Rietz, *Ann.*, 1907, 356, 164 (Note).

### 5-Nitro-6-hydroxy-2 : 3-dimethylbenzaldehyde



$\text{C}_9\text{H}_9\text{O}_4\text{N}$

MW, 195

Dark yellow needles from EtOH. M.p. 86–7°.

Clayton, *J. Chem. Soc.*, 1910, 97, 1405.

### 3-Nitro-4-hydroxy-2 : 5-dimethylbenzaldehyde.

Plates from EtOH. M.p. 188°.

**5-Nitro-6-hydroxy-3 : 4-dimethylbenz- 166**  
aldehyde

Oxime : orange-red needles from H<sub>2</sub>O. Decomp. at 160°.

Azine : orange needles from EtOH. Decomp. at 237°.

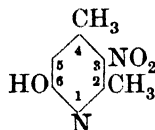
Gattermann, *Ann.*, 1907, **357**, 325.

**5-Nitro-6-hydroxy-3 : 4-dimethylbenz-aldehyde.**

Needles from EtOH. M.p. 146–7°.

Clayton, *J. Chem. Soc.*, 1910, **97**, 1405.

**3-Nitro-6-hydroxy-2 : 4-dimethylpyridine** (5-Nitro-4 : 6-dimethyl- $\alpha$ -pyridone, 3-nitro-6-hydroxy- $\alpha$ -lutidine, 5-nitro- $\alpha$ -lutidone)



C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>

MW, 168

Leaflets. M.p. 260°. Sol. H<sub>2</sub>O.

Moir, *J. Chem. Soc.*, 1902, **81**, 116.

**5-Nitro-6-hydroxy-2 : 4-dimethylpyridine** (3-Nitro-4 : 6-dimethyl- $\alpha$ -pyridone, 5-nitro-6-hydroxy- $\alpha$ -lutidine, 3-nitro- $\alpha$ -lutidone).

Yellow needles from AcOH. M.p. 254°. Sol. alkalis with yellow col. Non-volatile in steam.

Collie, Tickle, *J. Chem. Soc.*, 1898, **73**, 231.

Moir, *J. Chem. Soc.*, 1902, **81**, 104.

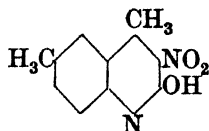
**3-Nitro-4-hydroxy-2 : 6-dimethylpyridine** (3-Nitro-2 : 6-dimethyl- $\gamma$ -pyridone, 3-nitro-4-hydroxy- $\alpha$ -lutidine, 3-nitro- $\gamma$ -lutidone).

Yellowish cryst. from AcOH. M.p. about 290–300° decomp. Sol. cold H<sub>2</sub>O. Sol. Na<sub>2</sub>CO<sub>3</sub>. Aq. with yellow col.

Hall, Collie, *J. Chem. Soc.*, 1898, **73**, 238.

Collie, Bishop, *J. Chem. Soc.*, 1925, **962**.

**3-Nitro-2-hydroxy-4 : 6-dimethylquinoline** (3-Nitro-4 : 6-dimethylcarbostyryl)



C<sub>11</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>

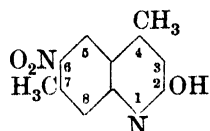
MW, 218

Prismatic needles from AcOH. M.p. 294°. Spar. sol. EtOH. Insol. H<sub>2</sub>O.

Balaban, *J. Chem. Soc.*, 1930, 2346.

**2'-Nitro-2-hydroxydiphenyl**

**6-Nitro-2-hydroxy-4 : 7-dimethylquinoline** (6-Nitro-4 : 7-dimethylcarbostyryl)



C<sub>11</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>

MW, 218

Leaflets from AcOH. Darkens at 280° with part. decomp. Spar. sol. AcOH. Insol. EtOH, Et<sub>2</sub>O.

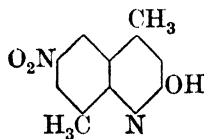
See previous reference.

**8-Nitro-2-hydroxy-4 : 7-dimethylquinoline** (8-Nitro-4 : 7-dimethylcarbostyryl).

Yellow plates from AcOH–EtOH. M.p. 226°. Sol. hot AcOH. Spar. sol. EtOH.

See previous reference.

**6-Nitro-2-hydroxy-4 : 8-dimethylquinoline** (6-Nitro-4 : 8-dimethylcarbostyryl)



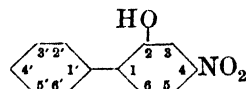
C<sub>11</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>

MW, 218

Needles from AcOH. M.p. 310° decomp. Spar. sol. AcOH.

See previous reference.

**4-Nitro-2-hydroxydiphenyl**



C<sub>12</sub>H<sub>9</sub>O<sub>3</sub>N

MW, 215

Cryst. from EtOH. M.p. 200–1°.

Finzi, Mangini, *Gazz. chim. ital.*, 1932, **62**, 672.

**5-Nitro-2-hydroxydiphenyl.**

Yellow needles from dil. EtOH or dil. AcOH. M.p. 128° (125–6°). Sol. EtOH, Me<sub>2</sub>CO. Mod. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. ligroin. Sublimes.

Me ether : C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 229. Yellow needles from MeOH. M.p. 95–6°.

Et ether : C<sub>14</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 243. M.p. 110–6°.

Borsche, Scholten, *Ber.*, 1917, **50**, 600.

Borsche, *Ann.*, 1900, **312**, 223.

Hill, *Ber.*, 1900, **33**, 1241.

**2'-Nitro-2-hydroxydiphenyl.**

Brown liq.

Me ether : m.p. 80–1°.

**4'-Nitro-2-hydroxydiphenyl**

167

 **$\beta$ -Nitro- $\alpha$ -hydroxy-*sym.*-diphenylethane***Acetyl*: cryst. from pet. ether. M.p. 102°.Mascarelli, Gatti, *Gazz. chim. ital.*, 1931, 61, 791; *Chem. Abstracts*, 1935, 29, 4351.**4'-Nitro-2-hydroxydiphenyl.**

Needles from dil. EtOH. M.p. 123-4°.

Harris, Christiansen, *Chem. Zentr.*, 1933, II, 3424.**3'-Nitro-3-hydroxydiphenyl.**

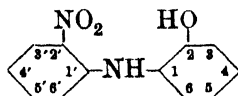
M.p. 114-16°.

Mascarelli, Gatti, *Gazz. chim. ital.*, 1931, 61, 325; *Chem. Abstracts*, 1930, 24, 4777.**3-Nitro-4-hydroxydiphenyl.**

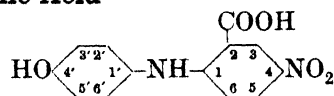
M.p. 66°.

*Me ether*: needles from EtOH. M.p. 91-2°.Raiford, Colbert, *J. Am. Chem. Soc.*, 1925, 47, 1454.Bell, Kenyon, *J. Chem. Soc.*, 1926, 3048.**2'-Nitro-4-hydroxydiphenyl.**

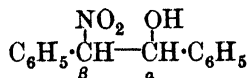
Yellow needles. M.p. 138°.

Schultz, Schmidt, Strasser, *Ann.*, 1881, 207, 351.**4'-Nitro-4-hydroxydiphenyl.**Yellow needles from  $C_6H_6$ . M.p. 203° (200-1°).*Me ether*: yellow needles from EtOH. M.p. 111°.*p-Toluenesulphonyl*: cryst. from  $C_6H_6$ . M.p. 159° (156-8°).Bell, Kenyon, *J. Chem. Soc.*, 1926, 3048.Angeletti, *Chem. Abstracts*, 1927, 21, 579.**2'-Nitro-2-hydroxydiphenylamine** $C_{12}H_{10}O_3N_2$ 

MW, 230

*Me ether*:  $C_{13}H_{12}O_3N_2$ . MW, 244. Red needles. M.p. 83°.McCombie, Scarborough, Waters, *J. Chem. Soc.*, 1928, 353.**6-Nitro-3-hydroxydiphenylamine.**Leaflets from  $C_6H_6$ . M.p. 165°. Sol. EtOH. Spar. sol.  $C_6H_6$ .*Et ether*:  $C_{14}H_{14}O_3N_2$ . MW, 258. Orange-yellow needles from EtOH. M.p. 106-106.5°.Jacobson, Fertsch, Fischer, *Ber.*, 1893, 26, 684.**2'-Nitro-4-hydroxydiphenylamine.***Me ether*: orange-red prisms. M.p. 89°.McCombie, Scarborough, Waters, *J. Chem. Soc.*, 1928, 353.**4'-Nitro-4-hydroxydiphenylamine.**Yellowish-brown leaflets with steel-blue reflex from  $H_2O$ . M.p. 183°. Sol. EtOH,  $Et_2O$ , AcOH, AcOEt. Spar. sol. boiling  $C_6H_6$ . Insol.  $CCl_4$ , ligroin. NaOH  $\rightarrow$  brown sol. Conc.  $H_2SO_4 \rightarrow$  pale green sol.  $\rightarrow$  blue on warming.Ullmann, D.R.P., 193,448, (*Chem. Zentr.*, 1908, I, 1003).Ullmann, Jüngel, *Ber.*, 1909, 42, 1078.**4-Nitro-4'-hydroxydiphenylamine-2-carboxylic Acid** $C_{13}H_{10}O_5N_2$ 

MW, 274

Orange powder. M.p. 210°. Excess NaOH.Aq.  $\rightarrow$  intense red col.Goldstein, Vaymatchar, *Helv. Chim. Acta*, 1928, 11, 243.**4'-Nitro-4-hydroxydiphenylamine-3-carboxylic Acid.**Brown needles. Sol. hot  $H_2O$  with orange-yellow col.M.L.B., D.R.P., 114,269, (*Chem. Zentr.*, 1900, II, 931).**2-Nitro-2'-hydroxydiphenylamine-4-carboxylic Acid.**Brown needles from EtOH.Aq. M.p. 260-1°. Very sol.  $Me_2CO$ . Sol. EtOH. Spar. sol.  $H_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , ligroin.Schöpf, *Ber.*, 1889, 22, 3288. **$\beta$ -Nitro- $\alpha$ -hydroxy-*sym.*-diphenylethane**  
( $\beta$ -Nitro- $\alpha$ -hydroxydibenzyl) $C_{14}H_{13}O_3N$ 

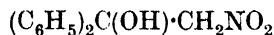
MW, 243

*Me ether*:  $C_{15}H_{15}O_3N$ . MW, 257. Exists in two forms. (i) Needles from EtOH or pet. ether. M.p. 130-1°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , alkalis. (ii) Prisms from MeOH. M.p. 97-8°. More sol. than (i).*Et ether*:  $C_{16}H_{17}O_3N$ . MW, 271. Needles. M.p. 92°. Mod. sol. usual solvents.Meisenheimer, Heim, *Ann.*, 1907, 355, 277.Heim, *Ber.*, 1911, 44, 2013.



**$\beta$ -Nitro- $\alpha$ -hydroxy-*unsym.*-diphenyl-ethane**

**$\beta$ -Nitro- $\alpha$ -hydroxy-*unsym.*-diphenyl-ethane**



$\text{C}_{14}\text{H}_{13}\text{O}_3\text{N}$

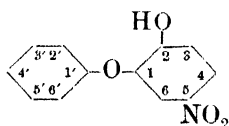
MW, 243

Prisms from AcOH. M.p. 107–8°. Sol. Et<sub>2</sub>O, hot AcOH. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether. Insol. H<sub>2</sub>O. CrO<sub>3</sub> in boiling AcOH  $\rightarrow$  benzophenone.

*Et ether*:  $\text{C}_{16}\text{H}_{17}\text{O}_3\text{N}$ . MW, 271. Cryst. from Et<sub>2</sub>O–pet. ether. M.p. 91–2°. Sol. Et<sub>2</sub>O, hot AcOH. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether.

Konowalow, Jatzewitsch, *Chem. Zentr.*, 1905, II, 825.

**5-Nitro-2-hydroxydiphenyl Ether**



$\text{C}_{12}\text{H}_9\text{O}_4\text{N}$

MW, 231

The structure given above has not been definitely established.

*Me ether*:  $\text{C}_{13}\text{H}_{11}\text{O}_4\text{N}$ . MW, 245. Needles from EtOH. M.p. 70–1°.

Lea, Robinson, *J. Chem. Soc.*, 1926, 412.

**2'-Nitro-2-hydroxydiphenyl Ether.**

*Me ether*: yellow needles. M.p. 55°. B.p. 213°/10 mm.

Bouveault, *Bull. soc. chim.*, 1897, 17, 949.

**3'-Nitro-2-hydroxydiphenyl Ether.**

*Me ether*: yellow prisms from MeOH. M.p. 86°.

Buchan, Scarborough, *J. Chem. Soc.*, 1934, 706.

**4'-Nitro-2-hydroxydiphenyl Ether.**

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 109°.

*Me ether*: yellow needles from EtOH. M.p. 106° (103–5–104°). B.p. 216°/10 mm.

See previous reference and also

Bouveault, *Bull. soc. chim.*, 1897, 17, 949.

**3-Nitro-4-hydroxydiphenyl Ether.**

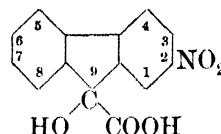
Yellow needles from pet. ether. M.p. 51–2°.

*Me ether*: pale yellow prisms from EtOH. M.p. 73–4°.

Lea, Robinson, *J. Chem. Soc.*, 1926, 412.

**168 o-Nitro- $\alpha$ -hydroxyhydrocinnamic Acid**

**2-Nitro-9-hydroxyfluorene-9-carboxylic Acid (2-Nitro-9-fluorenol-9-carboxylic acid)**



$\text{C}_{14}\text{H}_9\text{O}_5\text{N}$

MW, 271

Yellowish-brown prisms. M.p. 160–1° decomp. Sol. EtOH, MeOH, Me<sub>2</sub>CO. Mod. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. cold H<sub>2</sub>O. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with brownish-red col.  $\rightarrow$  green  $\rightarrow$  bluish-violet on warming.

Schmidt, Bauer, *Ber.*, 1905, 38, 3740.

**3-Nitro-9-hydroxyfluorene-9-carboxylic Acid (3-Nitro-9-fluorenol-9-carboxylic acid).**

Cryst. M.p. 239–40°.

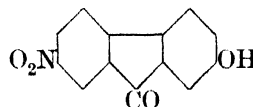
Schmidt, Söll, *Ber.*, 1908, 41, 3691.

**4-Nitro-9-hydroxyfluorene-9-carboxylic Acid (4-Nitro-9-fluorenol-9-carboxylic acid).**

Needles from H<sub>2</sub>O. M.p. 156–8°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Spar. sol. cold H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  brownish-yellow sol.  $\rightarrow$  green  $\rightarrow$  blue on warming.

Schmidt, Bauer, *Ber.*, 1905, 38, 3741.

**7-Nitro-2-hydroxyfluorenone**



$\text{C}_{13}\text{H}_7\text{O}_4\text{N}$

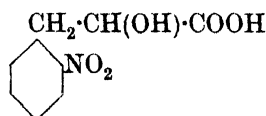
MW, 241

Red cryst. M.p. 298–9°.

*Me ether*:  $\text{C}_{14}\text{H}_9\text{O}_4\text{N}$ . MW, 255. M.p. 248–5–249–5°.

Eckert, Langecker, *J. prakt. Chem.*, 1928, 118, 275.

**o-Nitro- $\alpha$ -hydroxyhydrocinnamic Acid ( $\beta$ -2-Nitrophenyl-lactic acid, o-nitrobenzylglycollic acid,  $\alpha$ -hydroxy- $\beta$ -2-nitrophenylpropionic acid)**



$\text{C}_9\text{H}_9\text{O}_5\text{N}$

MW, 211

Needles from H<sub>2</sub>O. M.p. 72° (103°). Zn dust + HCl  $\rightarrow$  3-hydroxyhydrocarbostyryl.

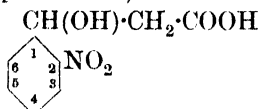
*Amide*:  $\text{C}_9\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 210. Needles from H<sub>2</sub>O. M.p. 195°. Sol. EtOH, Me<sub>2</sub>CO, hot H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

**o-Nitro-β-hydroxyhydrocinnamic Acid** 169

*Nitrile*:  $C_9H_8O_3N_2$ . MW, 192. Plates from  $CHCl_3$ -pet. ether. M.p. 70–1°.

Heller, *J. prakt. Chem.*, 1923, **106**, 1.  
Jaenisch, *Ber.*, 1923, **56**, 2450.

**o-Nitro-β-hydroxyhydrocinnamic Acid**  
(β-2-Nitrophenylhydracrylic acid, β-hydroxy-β-2-nitrophenylpropionic acid)



$C_9H_8O_5N$  MW, 211

Plates from  $H_2O$ , prisms from  $Et_2O$ . M.p. 126°. Sol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ . Dil.  $H_2SO_4$  at 190° → o-nitrocinnamic acid.

*Me ester*:  $C_{10}H_{11}O_5N$ . MW, 225. Cryst. from very dil.  $MeOH$ . M.p. 51°.

*Amide*:  $C_9H_{10}O_4N_2$ . MW, 210. Needles from  $EtOH$ . M.p. 197°, melting to give a blue col. Sol.  $H_2O$ ,  $EtOH$ ,  $AcOH$ . Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ , ligroin. *O-Acetyl*: prisms from  $EtOH$ . M.p. 141–2°.

Baeyer, Drewsen, *Ber.*, 1883, **16**, 2206.

Einhorn, *ibid.*, 2214.

Einhorn, Prausnitz, *Ber.*, 1884, **17**, 1660.

**m-Nitro-β-hydroxyhydrocinnamic Acid**  
(β-3-Nitrophenylhydracrylic acid, β-hydroxy-β-3-nitrophenylpropionic acid).

Leaflets from  $H_2O$ . M.p. 105°.

*Et ester*:  $C_{11}H_{13}O_5N$ . MW, 239. Cryst. from  $EtOH$ . Aq. M.p. 56°.

See last reference above and also  
Prausnitz, *Ber.*, 1884, **17**, 596, 598.

**p-Nitro-β-hydroxyhydrocinnamic Acid**  
(β-4-Nitrophenylhydracrylic acid, β-hydroxy-β-4-nitrophenylpropionic acid).

Cryst. M.p. 130–2°. Sol.  $EtOH$ ,  $Et_2O$ , ligroin, hot  $H_2O$ . Spar. sol.  $C_6H_6$ , cold  $H_2O$ . Sol. undecomp. in cold conc.  $H_2SO_4$ . Hot dil.  $H_2SO_4$  → p-nitrocinnamic acid.

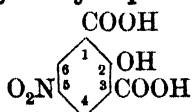
*Me ester*: cryst. from  $MeOH$ . Aq. M.p. 73–4°. Sol.  $EtOH$ ,  $Et_2O$ .

*Et ester*: cryst. from  $EtOH$ . Aq. M.p. 45–6°.

*Amide*: leaflets from  $H_2O$ . M.p. 166°.  
*O-Acetyl*: leaflets. M.p. 146–50°.

Basler, *Ber.*, 1883, **16**, 3003; 1884, **17**, 1494.

Einhorn, Prausnitz, *Ber.*, 1884, **17**, 1661.

**5-Nitro-2-hydroxyisophthalic Acid**

$C_8H_5O_7N$

MW, 227

**1-Nitro-4-hydroxy-2-methylantraquinone**

Needles +  $H_2O$ . M.p. anhyd. 213–14°. Sol. hot  $H_2O$ ,  $EtOH$ , hot  $AcOH$ . Spar. sol.  $Et_2O$ . Insol.  $C_6H_6$ ,  $CHCl_3$ .

*Mononitrile*:  $C_8H_4O_5N_2$ . MW, 208. Yellow cryst. +  $H_2O$  from  $H_2O$ . M.p. 205°. Sol.  $H_2O$ . Very spar. sol.  $EtOH$ . Oxidises readily in air. Difficult to hydrolyse.

Hill, *Am. Chem. J.*, 1900, **24**, 13.

Meldola, Foster, Brightman, *J. Chem. Soc.*, 1917, **111**, 545.

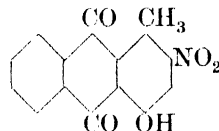
**5-Nitro-4-hydroxyisophthalic Acid.**

Cryst. from  $EtOH$ .

Chem. Fab. von Heyden, D.R.P., 555,410,  
(*Chem. Abstracts*, 1932, **26**, 5105).

**Nitrohydroxylepidine.**

See Nitrohydroxy-4-methylquinoline.

**2-Nitro-4-hydroxy-1-methylantraquinone**

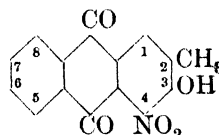
$C_{15}H_9O_5N$

MW, 283

Orange-yellow needles or prisms from  $EtOH$ . M.p. 182°. Turns red in air. Alkalis → red-dish-violet sols. Conc.  $H_2SO_4$  → orange-red sol.

Fischer, Rebsamen, *Ber.*, 1914, **47**, 465.

Fischer, Schweckendiek, *ibid.*, 1577.

**4-Nitro-3-hydroxy-2-methylantraquinone**

$C_{15}H_9O_5N$

MW, 283

Prisms from  $AcOH$ . M.p. 267°.

*Me ether*:  $C_{16}H_{11}O_5N$ . MW, 297. Plates from  $AcOH$ . M.p. 206°.

Mitter, Pal, *J. Indian Chem. Soc.*, 1930, **7**, 259.

**1-Nitro-4-hydroxy-2-methylantraquinone.**

Pale yellow needles from  $Me_2CO$ . M.p. 274–5° (241–2°). Sol.  $EtOH$ ,  $Me_2CO$ , toluene. Insol.  $H_2O$ .

Eder, Manoukian, *Helv. Chim. Acta*, 1926, **9**, 58.

Keimatsu, Hirano, *Chem. Abstracts*, 1932, 1601.

**3-Nitro-4-hydroxy-2-methylantraquinone**

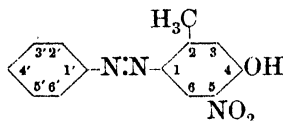
170

**3-Nitro-4-hydroxy-2-methylantraquinone.**

Pale yellow plates from  $\text{Me}_2\text{CO}$ . M.p. 272–3°.

See first reference above.

**5-Nitro-4-hydroxy-2-methylazobenzene**



$\text{C}_{13}\text{H}_{11}\text{O}_3\text{N}_3$

MW, 257

Yellowish-brown cryst. from pet. ether. M.p. 83–5°. Sol. most org. solvents.

Auwers, Michaelis, *Ber.*, 1914, **47**, 1296.

**4'-Nitro-4-hydroxy-2-methylazobenzene.**

Violet-brown needles with bronze reflex from toluene, orange-red needles from EtOH.Aq. M.p. 162.5–163.5°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. sol. cold  $\text{C}_6\text{H}_6$ , ligroin.

*Me ether*:  $\text{C}_{14}\text{H}_{13}\text{O}_3\text{N}_3$ . MW, 271. Orange-red needles and plates from EtOH. M.p. 138°. Sol.  $\text{C}_6\text{H}_6$ . Mod. sol. EtOH, AcOH. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  red sol.

Bamberger, *Ber.*, 1895, **28**, 847.

See also previous reference.

**3'-Nitro-4'-hydroxy-2-methylazobenzene.**

Cryst. from AcOH. M.p. 146°. Very sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Sol. AcOH. Spar. sol. EtOH.

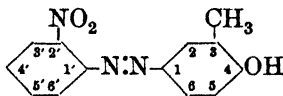
*Et ether*:  $\text{C}_{15}\text{H}_{15}\text{O}_3\text{N}_3$ . MW, 285. Orange needles from AcOH. M.p. 83°. Sol. EtOH.

*Acetyl*: golden-yellow needles from AcOH. M.p. 108°.

*Benzoyl*: cryst. from  $\text{C}_6\text{H}_6$ . M.p. 118°.

Hewitt, Lindfield, *J. Chem. Soc.*, 1901, **79**, 156.

**2'-Nitro-4-hydroxy-3-methylazobenzene.**



$\text{C}_{13}\text{H}_{11}\text{O}_3\text{N}_3$

MW, 257

Orange-red cryst. from  $\text{C}_6\text{H}_6$  or MeOH.Aq. M.p. 111–12°.

Borsche, *Ann.*, 1907, **357**, 177.

**4'-Nitro-4-hydroxy-3-methylazobenzene.**

Yellowish-brown cryst. from EtOH. M.p. 202°. Sol.  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , ligroin.

Bamberger, *Ber.*, 1895, **28**, 846.

**3-Nitro-4'-hydroxy-4-methylazobenzene**

**2'-Nitro-6-hydroxy-3-methylazobenzene.**

Red needles from EtOH. M.p. 118°.

*Acetyl*: yellow needles from EtOH. M.p. 99–100°.

Goldschmidt, Brubacher, *Ber.*, 1891, **24**, 2308.

**3'-Nitro-6-hydroxy-3-methylazobenzene.**

Brown needles from  $\text{C}_6\text{H}_6$ . M.p. 160–1°.

*Et ether*:  $\text{C}_{15}\text{H}_{15}\text{O}_3\text{N}_3$ . MW, 285. Orange needles from EtOH- $\text{C}_6\text{H}_6$ . M.p. 121–2°.

*Acetyl*: orange prisms from  $\text{C}_6\text{H}_6$ . M.p. 143–4°.

Meldola, Hanes, *J. Chem. Soc.*, 1894, **65**, 839.

**4'-Nitro-6-hydroxy-3-methylazobenzene.**

Brown needles from EtOH, leaflets from AcOH. M.p. 186.5°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, AcOH, ligroin.

*Acetyl*: red needles from EtOH. M.p. 184°. Sol. most org. solvents. Less sol. EtOH, pet. ether.

Mehner, *J. prakt. Chem.*, 1902, **65**, 453.

Auwers, *Ann.*, 1909, **365**, 310.

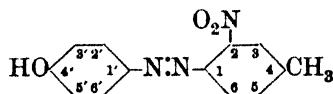
**3'-Nitro-4'-hydroxy-3-methylazobenzene.**

Yellow needles from pet. ether. M.p. 128.5°.

*Et ether*: brown cryst. from EtOH. M.p. 92°.

Hewitt, Lindfield, *J. Chem. Soc.*, 1901, **79**, 157.

**2-Nitro-4'-hydroxy-4-methylazobenzene**



$\text{C}_{13}\text{H}_{11}\text{O}_3\text{N}_3$

MW, 257

Brown cryst. from  $\text{C}_6\text{H}_6$ . M.p. 158°.

Hewitt, Mitchell, *J. Chem. Soc.*, 1905, **87**, 232.

**3-Nitro-4'-hydroxy-4-methylazobenzene.**

Orange cryst. from AcOH. M.p. 186°. Sol. AcOH. Mod. sol.  $\text{C}_6\text{H}_6$ ,  $\text{Et}_2\text{O}$ . Spar. sol. EtOH.

*Acetyl*: golden-yellow needles from AcOH.Aq. M.p. 113°. Very sol. EtOH,  $\text{C}_6\text{H}_6$ , AcOH.

Hewitt, Mitchell, *J. Chem. Soc.*, 1905, **87**, 231.

**3'-Nitro-4'-hydroxy-4-methylazo-benzene**

**3'-Nitro-4'-hydroxy-4-methylazobenzene.**

Brown plates from AcOH. M.p. 174°. Sol. most ord. solvents. Spar. sol. pet. ether.

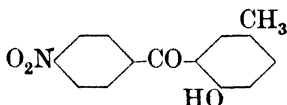
*Ether*:  $C_{15}H_{15}O_3N_3$ . MW, 285. Brownish needles from EtOH. M.p. 118°.

*Acetyl*: brown prisms from EtOH. M.p. 94°.

*Benzoyl*: yellow cryst. from EtOH. M.p. 129°.

Hewitt, Lindfield, *J. Chem. Soc.*, 1901, **79**, 158.

**4'-Nitro-6-hydroxy-3-methylbenzophenone**



$C_{14}H_{11}O_4N$

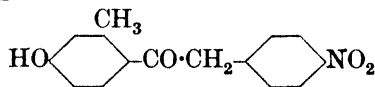
MW, 257

Yellow plates from EtOH. M.p. 142-3°. Sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH, ligroin.

*Me ether*:  $C_{15}H_{13}O_4N$ . MW, 271. Leaflets from ligroin. M.p. 101-2°.

Auwers, Rietz, *Ber.*, 1907, **40**, 3518.

**4-Nitro-4'-hydroxy-2'-methyldeoxybenzoin**



$C_{15}H_{13}O_4N$

MW, 271

Long needles from ligroin. M.p. 128°.

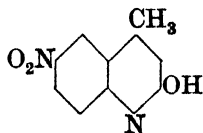
*Me ether*:  $C_{16}H_{15}O_4N$ . MW, 285. Plates from ligroin. M.p. 92-3°. *Oxime*: needles. M.p. 164.5-165°.

Hill, Short, *J. Chem. Soc.*, 1935, 1126.

**Nitrohydroxy-2-methylquinoline.**

See Nitrohydroxyquinaldine.

**6-Nitro-2-hydroxy-4-methylquinoline** (6-Nitro-4-methylcarbostyryl, 6-nitro-2-hydroxy-lepidine, 6-nitro-4-methyl-2-quinolinol)



$C_{10}H_8O_3N_2$

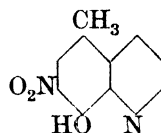
MW, 204

Prisms from AcOH. M.p. 340° decomp. Insol.  $H_2O$ , EtOH.  $KMnO_4 \rightarrow$  nitrobenzoxazolone.

Balaban, *J. Chem. Soc.*, 1930, 2349.

**171 5-Nitro-8-hydroxy-7-methylquinoline**

**7-Nitro-8-hydroxy-5-methylquinoline** (7-Nitro-5-methyl-8-quinolinol)



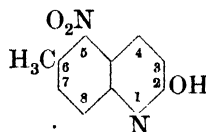
$C_{10}H_8O_3N_2$

MW, 204

Yellow needles from EtOH. M.p. 205-6°. Spar. sol.  $H_2O$ .

Noelting, Trautmann, *Ber.*, 1890, **23**, 3667.

**5-Nitro-2-hydroxy-6-methylquinoline** (5-Nitro-6-methylcarbostyryl, 5-nitro-6-methyl-2-quinolinol)



$C_{10}H_8O_3N_2$

MW, 204

*N-Me*: *N*-methyl- $\alpha$ -quinolone.  $C_{11}H_{10}O_3N_2$ . MW, 218. Cryst. from EtOH. M.p. 192°. Sol. HCl.

Decker, *J. prakt. Chem.*, 1892, **45**, 177.

**8-Nitro-2-hydroxy-6-methylquinoline** (8-Nitro-6-methylcarbostyryl, 8-nitro-6-methyl-2-quinolinol).

Yellow plates from EtOH. M.p. 200-1°.

*N-Me*: *N*-methyl- $\alpha$ -quinolone. Yellow needles from MeOH. M.p. 165-6°.

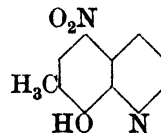
Ing, Cahn, *J. Chem. Soc.*, 1931, 2202.

**8-Nitro-5-hydroxy-6-methylquinoline** (8-Nitro-6-methyl-5-quinolinol).

Yellowish-brown leaflets from EtOH or AcOH. Decomp. on heating.

Noelting, Trautmann, *Ber.*, 1890, **23**, 3662.

**5-Nitro-8-hydroxy-7-methylquinoline** (5-Nitro-7-methyl-8-quinolinol)



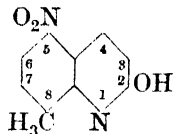
$C_{10}H_8O_3N_2$

MW, 204

Red needles from EtOH, yellow needles from  $C_6H_6$ . M.p. 192-3°. Spar. sol.  $H_2O$ .

Noelting, Trautmann, *Ber.*, 1890, **23**, 3665.

**5-Nitro-2-hydroxy-8-methylquinoline** (5-Nitro-8-methylcarbostyryl, 5-nitro-8-methyl-2-quinolinol)



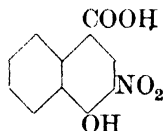
$C_{10}H_8O_3N_2$  MW, 204  
N-Me: N-methyl- $\alpha$ -quinolone. Yellow needles. M.p. 139°.

Decker, *Ber.*, 1905, **38**, 1153.

**6-Nitro-5-hydroxy-8-methylquinoline** (6-Nitro-8-methyl-5-quinolinol).

Orange-red needles from EtOH. M.p. 181-2°. Noelting, Trautmann, *Ber.*, 1890, **23**, 3677.

**3-Nitro-4-hydroxy-1-naphthoic Acid**

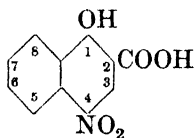


$C_{11}H_7O_5N$  MW, 233

Yellow needles from AcOH. M.p. 258° decomp. Very spar. sol.  $H_2O$  with yellow col. Sol.  $Na_2CO_3$ . Aq. with red col.

Heller, *Ber.*, 1912, **45**, 676.

**4-Nitro-1-hydroxy-2-naphthoic Acid**



$C_{11}H_7O_5N$  MW, 233

Yellow needles from EtOH.Aq. M.p. 214° decomp.  $H_2O$  at 150°  $\rightarrow$  4-nitro-1-naphthol.

Me ether:  $C_{12}H_9O_5N$ . MW, 247. Yellow leaflets from AcOH. M.p. 195-6°.

König, *Ber.*, 1890, **23**, 807.

Borsche, Berkhout, *Ann.*, 1904, **330**, 103.

Dey, *J. Chem. Soc.*, 1915, **107**, 1625.

Froehlicher, Cohen, *J. Chem. Soc.*, 1922, **121**, 1657.

**4-Nitro-3-hydroxy-2-naphthoic Acid.**

Golden-yellow prisms. M.p. 233-8° decomp. Sol. MeOH, EtOH. Spar. sol. AcOH. Very spar. sol.  $H_2O$ .

Me ester:  $C_{12}H_9O_5N$ . MW, 247. Yellow leaflets from  $Ac_2O$ . M.p. 189°. Alk.  $KMnO_4 \rightarrow$  phthalic acid.

Et ester:  $C_{13}H_{11}O_5N$ . MW, 261. Greenish

needles. M.p. 160° (156°). Spar. sol. EtOH, AcOH.

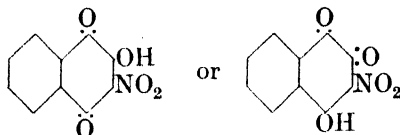
Acetyl: m.p. 235°. Anilide: m.p. 236-8°.

Robertson, *J. prakt. Chem.*, 1893, **48**, 534.

Gradenwitz, *Ber.*, 1894, **27**, 2623.

Meisenheimer, Theilacker, Beisswenger, *Ann.*, 1932, **495**, 275.

**3-Nitro-2-hydroxy-1:4-naphthoquinone** (3-Nitro-4-hydroxy-1:2-naphthoquinone)



$C_{10}H_5O_5N$  MW, 219

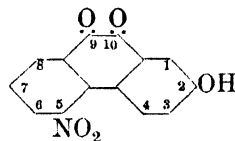
Yellow leaflets from  $CHCl_3$ . M.p. 157° decomp. Sol. EtOH,  $Et_2O$ , hot  $H_2O$ . Spar. sol.  $C_6H_6$ ,  $CHCl_3$ , ligroin. Dil.  $HNO_3 \rightarrow$  phthalic acid.

A.G.F.A., D.R.P., 100,611.

Diehl, Merz, *Ber.*, 1878, **11**, 1317.

Kehrman, *Ber.*, 1888, **21**, 1780.

**5-Nitro-2-hydroxyphenanthraquinone**



$C_{14}H_7O_5N$  MW, 269

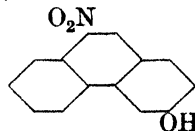
Reddish-brown needles from EtOH.Aq. M.p. about 240° decomp. Sol. EtOH,  $Et_2O$ , AcOEt with deep red col. Spar. sol.  $C_6H_6$ , AcOH. Sol. conc.  $H_2SO_4$  and alkalis with reddish-brown col.

Acetyl: greenish-yellow needles. M.p. about 220° decomp.

Christie, Holderness, Kenner, *J. Chem. Soc.*, 1926, 671.

Kuhn, Albrecht, *Ann.*, 1927, **455**, 281.

**9-Nitro-3-hydroxyphenanthrene** (9-Nitro-3-phenanthranol)



$C_{14}H_9O_3N$  MW, 239

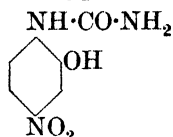
Yellow needles from toluene. M.p. 188-9°.

Acetyl: yellow needles from  $Me_2CO$ . M.p. 159°.

Burger, Mosettig, *J. Am. Chem. Soc.*, 1934, **56**, 1745.

**Nitrohydroxyphenylcinnamic Acid.**

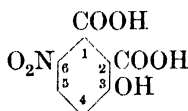
See Nitrohydroxystilbene-carboxylic Acid.

**4-Nitro-2-hydroxyphenylurea** $C_7H_7O_4N_3$ 

MW, 197

Cryst. from MeOH. M.p.  $203^\circ$  ( $205^\circ$ ). Sol. EtOH. Insol. cold  $H_2O$ ,  $Et_2O$ , acids. Alkalis  $\rightarrow$  red sols. Alc.  $FeCl_3 \rightarrow$  olive-green col. Above m.p.  $\rightarrow$  6-nitrobenzoxazolone.

Semper, Lichtenstadt, *Ann.*, 1913, **400**, 324.

**6-Nitro-3-hydroxyphthalic Acid** $C_8H_5O_7N$ 

MW, 227

*Dinitrile*:  $C_8H_3O_3N$ . MW, 161. *Me ether*:  $C_9H_5O_3N$ . MW, 175. M.p.  $198^\circ$ . *Et ether*:  $C_{10}H_7O_3N$ . MW, 189. M.p.  $160^\circ$ .

Blanksma, *Chem. Zentr.*, 1912, II, 339.

**3-Nitro-4-hydroxyphthalic Acid.**

*Me ether*:  $C_9H_7O_7N$ . MW, 241. Needles from  $Et_2O$ -Aq. M.p.  $215-17^\circ$  ( $212^\circ$ ) decomp. Mod. sol.  $H_2O$ , EtOH, AcOEt. Spar. sol.  $CHCl_3$ ,  $C_6H_6$ , pet. ether. *Di-Et ester*:  $C_{13}H_{15}O_7N$ . MW, 297. Needles from EtOH. M.p.  $93-4^\circ$ .

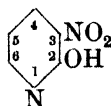
Cain, Simonsen, *J. Chem. Soc.*, 1914, **105**, 162.

Fischer, Kern, *J. prakt. Chem.*, 1916, **94**, 42.

**5-Nitro-4-hydroxyphthalic Acid.**

*Me ether*: prisms from  $H_2O$ . M.p.  $201^\circ$  decomp. *Di-Me ester*: needles from EtOH. M.p.  $115^\circ$ .

See first reference above.

**3-Nitro-2-hydroxypyridine** $C_5H_4O_3N_2$ 

MW, 140

*Me ether*:  $C_6H_6O_3N_2$ . MW, 154. M.p.  $110^\circ$ . Sol. EtOH,  $Et_2O$ .

Magidson, Menshikov, *Chem. Abstracts*, 1929, **23**, 1640.

**5-Nitro-2-hydroxypyridine.**

Yellow needles from  $H_2O$ . M.p.  $184^\circ$ . Sol. hot  $H_2O$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $CS_2$ , ligroin. Sol. alkalis. No col. with  $FeCl_3$ .

*Me ether*:  $C_6H_6O_3N_2$ . MW, 154. Needles from  $H_2O$ . M.p.  $110^\circ$ . Easily sol.  $Et_2O$ , EtOH.

*Et ether*:  $C_7H_8O_3N_2$ . MW, 168. M.p.  $95^\circ$  ( $72^\circ$ ).

*Butyl ether*:  $C_9H_{12}O_3N_2$ . MW, 196. B.p.  $148-50^\circ/22$  mm.

*Benzyl ether*:  $C_{12}H_{10}O_3N_2$ . MW, 230. M.p.  $107-8^\circ$ .

$\beta$ -Methoxyethyl ether: b.p.  $160-5^\circ/22$  mm. M.p.  $65-6^\circ$ .

*N-Me-pyridone*:  $C_6H_6O_3N_2$ . MW, 154. Cryst. from EtOH- $Et_2O$ . M.p.  $172^\circ$ .

Tschitschibabin, *J. Russ. Phys.-Chem. Soc.*, 1914, **46**, 1236.

Chemische Fabrik. von Heyden, D.R.P., 568,549, (*Chem. Zentr.*, 1933, I, 3468).

Pieroni, *Atti accad. Lincei*, 1927, **5**, 303.

Räth, E.P., 288,628, (*Chem. Abstracts*, 1929, **23**, 607).

Räth, *Ann.*, 1930, **484**, 52.

**6-Nitro-3-hydroxypyridine.**

*Et ether*: pale yellow needles from EtOH. M.p.  $31-2^\circ$ . B.p.  $175^\circ/50$  mm. Sol.  $Et_2O$ . Mod. sol. EtOH. Spar. sol.  $H_2O$ .

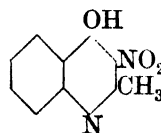
Koenigs, Gerdes, Sirot, *Ber.*, 1928, **61**, 1022.

**3-Nitro-4-hydroxypyridine.**

Yellow plates or needles. M.p.  $269-70^\circ$  decomp. Sol. hot  $H_2O$ , hot EtOH. Insol.  $Et_2O$ ,  $C_6H_6$ .

*Et ether*: needles. M.p.  $49-50^\circ$ . Easily sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ . *B.HCl*: needles. M.p.  $160^\circ$ . *Chloroplatinate*: needles. M.p.  $246-8^\circ$  decomp.

Koenigs, Freter, *Ber.*, 1924, **57**, 1188.

**3-Nitro-4-hydroxyquinaldine** $C_{10}H_8O_3N_2$ 

MW, 204

Needles from AcOH. Decomp. at  $280-5^\circ$ . Spar. sol. boiling EtOH,  $C_6H_6$ . Sol. conc. min. acids.

*Me ether*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Pale yellow needles from  $H_2O$  or 50% AcOH. M.p.  $192^\circ$ .

N-Oxide: decomp. at 227°. Sol. NaOH → orange-red col.

Conrad, Limpach, *Ber.*, 1887, 20, 950; 1888, 21, 1971.

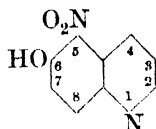
Stark, *Ber.*, 1907, 40, 3432.

Gabriel, Gerhard, *Ber.*, 1921, 54, 1076.

### Nitro-2-hydroxyquinoline.

See Nitrocarbostyryl.

### 5-Nitro-6-hydroxyquinoline



$C_9H_6O_3N_2$

MW, 190

Needles. M.p. 139–40°. Sol. hot EtOH, alkalis, min. acids.  $FeCl_3$  → red col. Sublimes.

*Me ether*:  $C_{10}H_8O_3N_2$ . MW, 204. Cryst. from EtOH. M.p. 104–5°. *B.HCl*: cryst. M.p. 219°. *B.HNO\_3*: m.p. 195°. Sol. warm EtOH. Insol.  $H_2O$ . *B\_2H\_2SO\_4*: m.p. 205°. *Picrate*: m.p. 211°. Sol.  $H_2O$ , EtOH. *Methiodide*: cryst. from  $H_2O$ . M.p. 275°. Sol. warm  $H_2O$ . Insol. EtOH. *Methiopicate*: yellow needles from EtOH. M.p. 186°.

*Et ether*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Prisms or needles. M.p. 111°. Sol. 20 parts cold EtOH. Sweet taste. *B.HNO\_3*: m.p. 193°. Insol. cold  $H_2O$ .

Vis, *J. prakt. Chem.*, 1893, 48, 27.

Decker, Engler, *Ber.*, 1909, 42, 1740.

Claus, Hofmann, *J. prakt. Chem.*, 1897, 55, 519.

### 8-Nitro-6-hydroxyquinoline.

Cryst. M.p. 230° decomp.

*Me ether*: cryst. M.p. 159–60°.

*Et ether*: m.p. 158°.

*Propyl ether*:  $C_{12}H_{12}O_3N_2$ . MW, 232. Prisms. M.p. 89°.

*Butyl ether*:  $C_{13}H_{14}O_3N_2$ . MW, 246. M.p. 92°.

*Isoamyl ether*:  $C_{14}H_{16}O_3N_2$ . MW, 260. M.p. 83°.

*Octyl ether*:  $C_{17}H_{22}O_3N_2$ . MW, 302. Cryst. M.p. 61°.

*Allyl ether*:  $C_{12}H_{10}O_3N_2$ . MW, 230. M.p. 114–5°.

*Phenyl ether*:  $C_{15}H_{10}O_3N_2$ . MW, 266. M.p. 142°.

Bayer, E.P., 267,457, (*Chem. Abstracts*, 1928, 22, 1216).

Magidson, Strukov, *Arch. Pharm.*, 1933, 271, 359.

### 8-Nitro-7-hydroxyquinoline.

Yellow plates. M.p. 255° decomp.  $FeCl_3$  in EtOH.Aq. → reddish-yellow col.

*Me ether*:  $C_{10}H_8O_3N_2$ . MW, 204. Pale yellow prisms from  $CHCl_3$ . M.p. 178°. *B.HNO\_3*: yellow needles. M.p. 155–6° decomp.

Skraup, *Monatsh.*, 1882, 3, 534.

Balaban, *J. Chem. Soc.*, 1932, 2626.

### 5-Nitro-8-hydroxyquinoline.

Yellow needles from EtOH or AcOH. M.p. 178°. Sol. AcOH, hot HCl. Spar. sol. EtOH,  $Et_2O$ . Sol. alkalis. Volatile in steam.

*B.HCl*: yellow needles from EtOH. M.p. 258°.

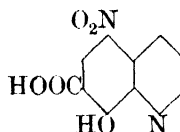
*Me ether*:  $C_{10}H_8O_3N_2$ . MW, 204. Cryst. from EtOH. M.p. 151–5°. Sol. hot EtOH. Insol.  $H_2O$ .

*Et ether*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Yellow needles. M.p. 128°. Sol. hot EtOH. Insol.  $H_2O$ . *B.HNO\_3*: needles. M.p. 100°. *B\_2H\_2PtCl\_6*: needles. Decomp. at 248°. *Methiodide*: red cryst. from EtOH, yellow cryst. + 2 $H_2O$  from  $H_2O$ . M.p. 150°.

Vis, *J. prakt. Chem.*, 1892, 45, 534.

Freyss, Paira, *Chem. Zentr.*, 1903, I, 35.

### 5-Nitro-8-hydroxyquinoline-7-carboxylic Acid



$C_{10}H_6O_5N_2$

MW, 234

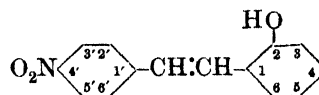
Yellow needles from HCl. Decomp. at 200°. Sol. conc. HCl, alkalis,  $NH_3$ . Spar. sol. AcOH. Glycerol at 200° → 5-nitro-8-hydroxyquinoline.

*Me ester*:  $C_{11}H_8O_5N_2$ . MW, 248. Needles from EtOH. M.p. 191°. Very sol.  $C_6H_6$ ,  $CHCl_3$ , AcOEt.

Einhorn, *Ann.*, 1900, 311, 65.

Schmitt, Engelmann, *Ber.*, 1887, 20, 2693.

### 4'-Nitro-2-hydroxystilbene



$C_{14}H_{11}O_3N$

MW, 241

*Me ether*:  $C_{15}H_{13}O_3N$ . MW, 255. Yellow needles from EtOH. M.p. 122°. Very sol.  $C_6H_6$ ,  $CHCl_3$ . Less sol. EtOH,  $Et_2O$ , AcOH.

Pfeiffer, *Ber.*, 1915, 48, 1795.

#### 4'-Nitro-3-hydroxystilbene

##### 4'-Nitro-3-hydroxystilbene.

*Me ether*: yellowish leaflets from EtOH. M.p. 87°. Very sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Sol. Et<sub>2</sub>O, AcOH. Less sol. EtOH.

See previous reference.

##### 2'-Nitro-4-hydroxystilbene.

*Me ether*: dark yellow needles from EtOH. M.p. 88-90°. Sol. EtOH, CHCl<sub>3</sub>.

See previous reference.

##### 4'-Nitro-4-hydroxystilbene.

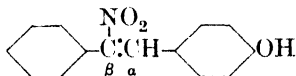
Yellow needles from EtOH.Aq. M.p. 193°. Alkalis → deep brown col.

*Me ether*: orange-yellow leaflets from AcOH; greenish-yellow needles from dil. C<sub>6</sub>H<sub>6</sub> sol. M.p. 132-4°. Greenish-yellow form → orange-yellow at 100°.

Hewitt, Lewcock, *J. Chem. Soc.*, 1931, 444.

See also previous reference.

##### β-Nitro-4-hydroxystilbene



C<sub>14</sub>H<sub>11</sub>O<sub>3</sub>N

MW, 241

*Me ether*: C<sub>15</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 255. Exists in two forms. (i) Golden-yellow needles from EtOH. M.p. 151°. Insol. Et<sub>2</sub>O. (ii) Brownish-orange needles. M.p. 112-13°.

Knoevenagel, Walter, *Ber.*, 1904, 37, 4509.

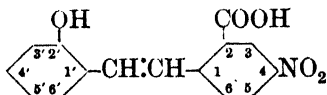
Flürscheim, Holmes, *J. Chem. Soc.*, 1932, 1463.

##### β-Nitro-α-hydroxystilbene.

*Me ether*: yellow solid. M.p. 87°.

Meisenheimer, Mahler, *Ann.*, 1934, 508, 190.

##### 4-Nitro-2'-hydroxystilbene-2-carboxylic Acid (5-Nitro-2-[o-hydroxystyryl]-benzoic acid)



C<sub>16</sub>H<sub>11</sub>O<sub>5</sub>N

MW, 285

*Me ether*: C<sub>16</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 299. *Nitrile*: C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 280. Yellow needles from AcOH. M.p. 146-8°. Sol. AcOH. Spar. sol. boiling EtOH. Very stable to alk. hydrolysis.

Pfeiffer, *Ber.*, 1916, 49, 2433.

#### 2-Nitro-3'-hydroxystilbene-4-carboxylic Acid

##### 4-Nitro-4'-hydroxystilbene-2-carboxylic Acid (5-Nitro-2-[p-hydroxystyryl]-benzoic acid).

*Me ether*: yellow needles + H<sub>2</sub>O from AcOH. M.p. 215°. Sol. AcOH, hot EtOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Insol. ligroin. *Amide*: C<sub>16</sub>H<sub>14</sub>O<sub>4</sub>N<sub>2</sub>. MW, 298. Yellow cryst. powder from xylene. M.p. 255°. *Nitrile*: m.p. 198°. Sol. Me<sub>2</sub>CO.

*Nitrile*: C<sub>15</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 266. Orange-yellow needles from AcOH. M.p. 226°. Sol. AcOH, boiling EtOH. KOH → red sol. *Acetyl*: yellow needles from AcOH. M.p. 176°. *Benzoyl*: yellow needles from AcOH. M.p. 178°.

Pfeiffer, *Ber.*, 1915, 48, 1796; 1916, 49, 2437; 1918, 51, 562.

##### 4-Nitro-4'-hydroxystilbene-3-carboxylic Acid (6-Nitro-3-[p-hydroxystyryl]-benzoic acid)



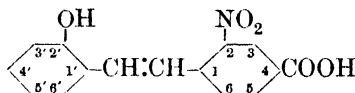
C<sub>15</sub>H<sub>11</sub>O<sub>5</sub>N

MW, 285

*Me ether*: C<sub>16</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 299. *Nitrile*: C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 280. Brownish-orange leaflets from AcOH. M.p. 161°. Sol. EtOH, hot AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin.

Pfeiffer, *Ber.*, 1918, 51, 561.

##### 2-Nitro-2'-hydroxystilbene-4-carboxylic Acid (3-Nitro-4-[o-hydroxystyryl]-benzoic acid)



C<sub>15</sub>H<sub>11</sub>O<sub>5</sub>N

MW, 285

*Me ether*: C<sub>16</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 299. Greenish-yellow cryst. from propionic acid. M.p. 230°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. *Et ester*: C<sub>18</sub>H<sub>17</sub>O<sub>5</sub>N. MW, 327. Yellow needles or leaflets from EtOH. M.p. 101°. Sol. AcOH, C<sub>6</sub>H<sub>6</sub>. *Nitrile*: C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 280. Yellow needles from AcOH. M.p. 183°. Sol. AcOH, C<sub>6</sub>H<sub>6</sub>.

Pfeiffer, *Ber.*, 1915, 48, 1803; 1916, 49, 2431.

##### 2-Nitro-3'-hydroxystilbene-4-carboxylic Acid (3-Nitro-4-[m-hydroxystyryl]-benzoic acid).

*Me ether*: greenish-yellow needles from AcOH. M.p. 240°. Sol. AcOH, C<sub>6</sub>H<sub>6</sub>. Very spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O, ligroin. *Nitrile*: greenish-yellow needles from AcOH. M.p. 163-4°. Sol. AcOH. Mod. sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, Et<sub>2</sub>O, ligroin.

Pfeiffer, *Ber.*, 1916, 49, 2434.



**2-Nitro-4'-hydroxystilbene-4-carboxylic Acid**

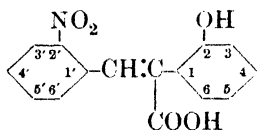
176

**3-Nitro-4-hydroxystyrene****2-Nitro-4'-hydroxystilbene-4-carboxylic Acid** (3-Nitro-4-[p-hydroxystyryl]-benzoic acid).

*Me ether*: exists in two forms. (i) Yellow needles or leaflets from AcOH or EtOH. (ii) Orange powder. At 140° (ii) → (i) and melts at 250°. Sol. hot AcOH. Spar. sol. hot H<sub>2</sub>O. *Me ester*: C<sub>17</sub>H<sub>15</sub>O<sub>5</sub>N. MW, 313. Yellow needles from MeOH. M.p. 117–18°. At 100–5° → orange form. *Et ester*: C<sub>18</sub>H<sub>17</sub>O<sub>5</sub>N. MW, 327. Yellow needles from EtOH. M.p. 103–4°. Sol. Me<sub>2</sub>CO, AcOH, Py, CCl<sub>4</sub>. *Nitrile*: yellow or orange cryst. from C<sub>6</sub>H<sub>6</sub> or AcOH. M.p. 157–8°. At 120–5° yellow → orange modification.

*Nitrile*: C<sub>15</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 266. M.p. 230–1°. *Acetyl*: yellow leaflets from AcOH. M.p. 225°.

Pfeiffer, *Ber.*, 1915, **48**, 1799; 1916, **49**, 2435; 1918, **51**, 564; *Ann.*, 1916, **411**, 144.

**2'-Nitro-2-hydroxystilbene-α-carboxylic Acid** (2-Nitro-α-[2-hydroxyphenyl]-cinnamic acid)C<sub>15</sub>H<sub>11</sub>O<sub>5</sub>N

MW, 285

*Me ether*: C<sub>16</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 299. Yellowish plates from EtOH. M.p. 219–20°. Spar. sol. ligroin. Insol. H<sub>2</sub>O.

Pschorr, Wolfes, Buckow, *Ber.*, 1900, **33**, 167.

**2'-Nitro-4-hydroxystilbene-α-carboxylic Acid** (2-Nitro-α-[4-hydroxyphenyl]-cinnamic acid).

*Me ether*: C<sub>16</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 299. Prisms from toluene, leaflets from EtOH. M.p. 177° decomp. Spar. sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O, ligroin. *Nitrile*: C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 280. Yellow needles from toluene or EtOH. M.p. 162°. Spar. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O, ligroin. Sublimes.

*Et ether*: C<sub>17</sub>H<sub>15</sub>O<sub>5</sub>N. MW, 313. Yellow prisms from AcOH or toluene. M.p. 158°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. ligroin. Insol. H<sub>2</sub>O.

Pschorr, Wolfes, *Ber.*, 1899, **32**, 3400.

Pschorr, Wolfes, Buckow, *Ber.*, 1900, **33**, 172.

Werner, *Ann.*, 1902, **322**, 152.

**4-Nitro-2'-hydroxystilbene-α-carboxylic Acid** (2-Hydroxy-α-[4-nitrophenyl]-cinnamic acid).

*Me ether*: C<sub>16</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 299. *Nitrile*: C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 280. Fluor. yellow needles from AcOH. M.p. 190°.

Kauffmann, *Ber.*, 1917, **50**, 1621.

**2'-Nitro-3'-hydroxystilbene-α-carboxylic Acid** (2-Nitro-3-hydroxy-α-phenylcinnamic acid).

*Me ether*: leaflets from EtOH.Aq. M.p. 226–7°. Sol. EtOH, CHCl<sub>3</sub>, AcOH. Spar. sol. Et<sub>2</sub>O, toluene. Insol. H<sub>2</sub>O, ligroin.

Pschorr, Jaeckel, *Ber.*, 1900, **33**, 1826.

**4'-Nitro-3'-hydroxystilbene-α-carboxylic Acid** (4-Nitro-3-hydroxy-α-phenylcinnamic acid).

*Acetyl*: yellowish prisms from EtOH. M.p. 254°.

Pschorr, *Ber.*, 1906, **39**, 3123.

**6'-Nitro-3'-hydroxystilbene-α-carboxylic Acid** (6-Nitro-3-hydroxy-α-phenylcinnamic acid).

Leaflets from EtOH.Aq. or AcOH.Aq. M.p. 219–20°.

*Me ether*: C<sub>16</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 299. Leaflets from EtOH. M.p. 165–6°. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin. Insol. H<sub>2</sub>O.

Pschorr, Seydel, *Ber.*, 1901, **34**, 4000.

See also previous reference.

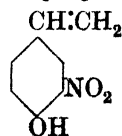
**4-Nitro-4'-hydroxystilbene-α-carboxylic Acid** (4-Hydroxy-α-[4-nitrophenyl]-cinnamic acid).

*Me ether*: orange cryst. powder from EtOH. M.p. 231°. Dist. under red. press. → 4'-nitro-4-methoxystilbene. *Nitrile*: C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 280. Yellow needles from EtOH. M.p. 165–6°. Sol. C<sub>6</sub>H<sub>6</sub>, AcOH.

*Acetyl*: yellowish cryst. powder from CCl<sub>4</sub>-AcOH. M.p. 205°. Heat with EtOH-conc. HCl or piperidine → 4'-nitro-4-hydroxystilbene.

Remse, *Ber.*, 1890, **23**, 3135.

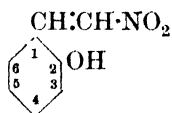
Hewitt, Lewcock, Pope, *J. Chem. Soc.*, 1912, **101**, 607.

**3-Nitro-4-hydroxystyrene**C<sub>8</sub>H<sub>7</sub>O<sub>3</sub>N

MW, 165

*Me ether*:  $C_9H_9O_3N$ . MW, 179. Cryst. from EtOH. M.p. 89°. Sol. usual org. solvents. Volatile in steam.

Einhorn, Grabfield, *Ann.*, 1888, **243**, 368.

 **$\beta$ -Nitro-*o*-hydroxystyrene**

$C_8H_7O_3N$

MW, 165

Yellowish needles. M.p. 133–4°.

Remfry, *J. Chem. Soc.*, 1911, **99**, 286.

 **$\beta$ -Nitro-*m*-hydroxystyrene.**

Yellowish needles. M.p. 132–3°.

*Me ether*:  $C_9H_9O_3N$ . MW, 179. Pale yellow plates or needles from EtOH. M.p. 93–4°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol. ligroin.

*Benzyl ether*: yellow needles from EtOH. M.p. 83°.

Kondo, Ishiwata, *Ber.*, 1931, **64**, 1538.

Gulland, Virden, *J. Chem. Soc.*, 1929, 1795.

Shoesmith, Connor, *J. Chem. Soc.*, 1927, 2232.

See also previous reference.

 **$\beta$ -Nitro-*p*-hydroxystyrene.**

Yellowish needles or plates from EtOH.Aq. M.p. 165°.

*Me ether*: anisylidenenitromethane. Yellow needles from EtOH, plates from  $C_6H_6$ . M.p. 88°. Sol. hot EtOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Sol. caustic alkalis with yellow col.

*Benzyl ether*: yellow cryst. from EtOH. M.p. 120°.

*Acetyl*: yellow needles from EtOH. M.p. 163° (158–9°).

Rosenmund, Pfannkuch, *Ber.*, 1922, **55**, 2365.

Kondo, Shinozaki, *J. Pharm. Soc. Japan*, 1929, **49**, 267.

Knoevenagel, Walter, *Ber.*, 1904, **37**, 4505.

Rosenmund, *Ber.*, 1909, **42**, 4779; 1913, **46**, 1041.

Remfry, *J. Chem. Soc.*, 1911, **99**, 286.

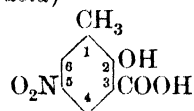
Bouveault, Wahl, *Bull. soc. chim.*, 1903, **29**, 523.

Knoevenagel, D.R.P., 161,171, (*Chem. Zentr.*, 1905, II, 179).

**Nitrohydroxystyrylbenzoic Acid.**

See Nitrohydroxystyrene-carboxylic Acid.

Dict. of Org. Comp.—III.

**5-Nitro-2-hydroxy-*m*-toluic Acid (5-Nitro-*o*-cresotic acid)**

$C_8H_7O_5N$

MW, 197

Needles from EtOH.Aq. M.p. 199°.

*Et ester*:  $C_{10}H_{11}O_5N$ . MW, 225. Leaflets from EtOH.Aq. M.p. 63–4°.

*Chloride*:  $C_8H_6O_4NCl$ . MW, 215.5. Cryst. from  $C_6H_6$ . M.p. 86–8°.

*Amide*:  $C_8H_8O_4N_2$ . MW, 196. Yellowish cryst. from EtOH. M.p. 231°. Spar. sol. EtOH, AcOH.

*Acetyl*: yellowish cryst. from EtOH.Aq. M.p. 142°. Sol. EtOH. Less sol.  $H_2O$ , AcOH.

Einhorn, Pfyl, *Ann.*, 1900, **311**, 47.

Fortner, *Monatsh.*, 1901, **22**, 944.

**5-Nitro-4-hydroxy-*m*-toluic Acid (5-Nitro-*p*-cresotic acid).**

Cryst. from AcOH. M.p. 173°.

*Et ester*: m.p. 104–5°. *p*-Toluenesulphonyl: plates from EtOH. M.p. 110°.

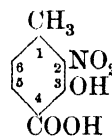
Sane, Chakravarty, Parmanick, *J. Indian Chem. Soc.*, 1932, **9**, 55.

**5-Nitro-6-hydroxy-*m*-toluic Acid.**

Pale yellow needles from AcOH or  $C_6H_6$ . M.p. 240°. Sol. EtOH,  $Et_2O$ , AcOH. Mod. sol. hot  $C_6H_6$ . Spar. sol.  $H_2O$ .

*Me ester*:  $C_9H_9O_5N$ . MW, 211. Yellow cryst. M.p. 103°.

Pfister, *J. Am. Chem. Soc.*, 1921, **43**, 375.

**2-Nitro-3-hydroxy-*p*-toluic Acid (2-Nitro-*m*-cresotic acid)**

$C_8H_7O_5N$

MW, 97

*Et ester*:  $C_{10}H_{11}O_5N$ . MW, 225. Needles from EtOH.Aq. M.p. 73–4°.

Einhorn, Pfyl, *Ann.*, 1900, **311**, 50.

**6-Nitro-3-hydroxy-*p*-toluic Acid (6-Nitro-*m*-cresotic acid).**

Needles from  $H_2O$ . M.p. 219° (213°).

*Me ether*:  $C_9H_9O_5N$ . MW, 211. Needles from  $H_2O$ . M.p. 173–5°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ .

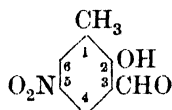
*Et ether*:  $C_{10}H_{11}O_5N$ . MW, 225. Needles from  $H_2O$ . M.p. 161–2°. Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Mod. sol. hot  $H_2O$ .

*Me ester*:  $C_9H_9O_5N$ . MW, 211. Cryst. from MeOH. M.p.  $78^\circ$ . *p*-Toluenesulphonyl: cryst. M.p.  $93^\circ$ .

Paternò, Canzoneri, *Gazz. chim. ital.*, 1879, 9, 456.

Clayton, *J. Chem. Soc.*, 1910, 97, 1402.

### 5-Nitro-2-hydroxy-*m*-toluic Aldehyde



$C_8H_7O_4N$

MW, 181

Yellow cryst. from AcOH, needles from ligroin. M.p.  $134^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Mod. sol. EtOH, AcOH. Spar. sol.  $H_2O$ , ligroin. Sublimes.

*Anil*: golden-yellow needles from  $C_6H_6$ . M.p.  $176-7^\circ$ . Sol.  $C_6H_6$ . Mod. sol. EtOH, AcOH. Spar. sol. ligroin.

*Phenylhydrazone*: yellow needles from EtOH. M.p.  $206-7^\circ$ .

Auwers, Bondy, *Ber.*, 1904, 37, 3916

### 5-Nitro-4-hydroxy-*m*-toluic Aldehyde.

Yellow needles. M.p.  $141^\circ$ . Spar. sol. hot  $H_2O$ .  $FeCl_3 \rightarrow$  violet col.

*Oxime*: light yellow leaflets from EtOH. M.p.  $214-16^\circ$ . Spar. sol. boiling EtOH.

*Anil*: orange-red needles from  $C_6H_6$ . M.p.  $133.5-134.5^\circ$ . Sol.  $C_6H_6$ . Mod. sol. EtOH, AcOH. Spar. sol.  $Et_2O$ , ligroin.

*Phenylhydrazone*: m.p.  $164-6^\circ$ .

Borsche, *Ber.*, 1917, 50, 1345.

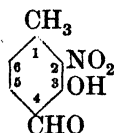
### 5-Nitro-6-hydroxy-*m*-toluic Aldehyde.

Yellowish needles from EtOH.Aq. M.p.  $152^\circ$ . Spar. sol. boiling  $H_2O$ . Volatile in steam. Forms bisulphite comp.

*Phenylhydrazone*: m.p.  $153-5^\circ$ .

Schotten, *Ber.*, 1878, 11, 789.

### 2-Nitro-3-hydroxy-*p*-toluic Aldehyde



$C_8H_7O_4N$

MW, 181

Yellow needles from EtOH. M.p.  $126-7^\circ$ .

Clayton, *J. Chem. Soc.*, 1910, 97, 1405.

### 6-Nitro-3-hydroxy-*p*-toluic Aldehyde.

Yellow needles from EtOH. M.p.  $144-5^\circ$ .

*Oxime*: yellow needles from EtOH. M.p.  $207-8^\circ$  decomp.

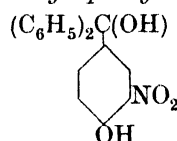
*Phenylhydrazone*: cryst. M.p.  $201-2^\circ$ .

See previous reference.

### Nitrohydroxytrimethylbenzene.

See Nitrohemimellitenol and Nitromesitol.

### 3-Nitro-4-hydroxytriphenylcarbinol (3-Nitro-4 : $\alpha$ -dihydroxytriphenylmethane)



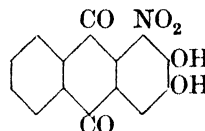
$C_{19}H_{15}O_4N$

MW, 321

Cryst. from 60% AcOH. M.p.  $97-8^\circ$ .

Gomberg, v. Stone, *J. Am. Chem. Soc.*, 1916, 38, 1604.

### 1-Nitrohystazarin (1-Nitro-2 : 3-dihydroxy-anthraquinone)



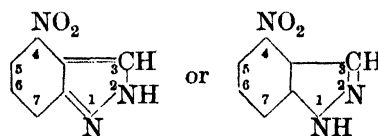
$C_{14}H_7O_6N$

MW, 285

Yellow cryst. from toluene. Sol. EtOH,  $Et_2O$ , AcOH. Less sol.  $H_2O$ ,  $C_6H_6$ . Dil. alkalis  $\rightarrow$  blue sols. Conc.  $H_2SO_4 \rightarrow$  orange-yellow sol.  $HNO_3 \rightarrow$  phthalic acid.

Schrobsdorff, *Ber.*, 1903, 36, 2939.

### 4-Nitroindazole



$C_7H_5O_2N_3$

MW, 163

Needles from  $H_2O$ . M.p.  $203^\circ$ . Very sol.  $Me_2CO$ , AcOH. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol. ligroin. Sol. alkalis with reddish-yellow col. Spar. volatile in steam.

*N-Me*: see 4-Nitro-methylindazole.

1-*o*-Tolyl:  $C_{14}H_{11}O_2N_3$ . MW, 253. Needles from EtOH.Aq. M.p.  $92-4^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ , AcOH. Mod. sol. EtOH, ligroin.

1-*p*-Tolyl: yellow needles from MeOH. M.p.  $155-6^\circ$ .

1-Benzyl:  $C_{14}H_{11}O_2N_3$ . MW, 253. Yellow needles from EtOH. M.p.  $97-8^\circ$ .

2-Benzyl: yellow needles from ligroin. M.p.  $123-4^\circ$ . Sol. most solvents.

*N-Acetyl*: (i) *Stable form*, needles from

MeOH. M.p. 144.5–145.5°. (ii) *Labile form*: yellow needles from Et<sub>2</sub>O. M.p. 119–21°.

N-Benzoyl: (i) *Stable form*, needles. M.p. 162–3°. (ii) *Labile form*: yellow needles from Et<sub>2</sub>O. M.p. 130–2°.

Noelting, *Ber.*, 1904, **37**, 2562.

Auwers, Schwegler, *Ber.*, 1920, **53**, 1211.

Auwers, Frese, *Ber.*, 1925, **58**, 1372.

### 5-Nitroindazole.

Leaflets or needles from EtOH. M.p. 208°. Very sol. Me<sub>2</sub>CO, AcOH. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. Spar. volatile in steam. Conc. alkalis → red sols. → yellow on dilution.

N-Acetyl: (i) *Stable form*, needles from EtOH. M.p. 158–9°. (ii) *Labile form*: needles from Me<sub>2</sub>CO–Et<sub>2</sub>O. M.p. 138–9°.

N-Benzoyl: (i) *Stable form*, leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 193–4°. (ii) *Labile form*: prisms from Me<sub>2</sub>CO. M.p. 155°.

N-Me: see 5-Nitro-methylindazole.

See first two references above.

### 6-Nitroindazole.

Needles from toluene. M.p. 181°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

N-Me: see 6-Nitro-methylindazole.

N-Et: C<sub>9</sub>H<sub>9</sub>O<sub>2</sub>N<sub>3</sub>. MW, 191. Needles from EtOH.Aq. M.p. 91–3°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, conc. HCl.

N-Benzyl: C<sub>14</sub>H<sub>11</sub>O<sub>2</sub>N<sub>3</sub>. MW, 253. Yellowish needles from MeOH. M.p. 111–12°.

N-Acetyl: (i) *Stable form*, needles. M.p. 140–1°. (ii) *Labile form*: leaflets from Et<sub>2</sub>O. M.p. 74–5°.

1-Chloroacetyl: yellowish needles from C<sub>6</sub>H<sub>6</sub>. M.p. 149°. Sol. hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold MeOH.

1-Dichloroacetyl: cryst. from 3 parts C<sub>6</sub>H<sub>6</sub> + 1 part pet. ether. M.p. 137–8°. Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>.

1-Trichloroacetyl: yellowish cryst. from C<sub>6</sub>H<sub>6</sub>–pet. ether. M.p. 101–2°.

N-Benzoyl: (i) *Stable form*, needles. M.p. 164–5°. (ii) *Labile form*: needles from Et<sub>2</sub>O. M.p. 133–4°.

1-o-Nitrobenzoyl: yellowish cryst. from toluene. M.p. 206–7°. Spar. sol. most solvents. Stable.

1-m-Nitrobenzoyl: cryst. from toluene. M.p. 212–13°. Spar. sol. most solvents. Stable.

1-p-Nitrobenzoyl: needles from PhNO<sub>2</sub>. M.p. 236–7°. Spar. sol. most solvents. Stable.

1-o-Toluyyl: yellowish needles from Me<sub>2</sub>CO. M.p. 188°. Mod. sol. Me<sub>2</sub>CO. Stable.

1-m-Toluyyl: yellowish needles from Me<sub>2</sub>CO. M.p. 159–60°.

1-p-Toluyyl: needles. M.p. 165–6°. Mod. sol. Me<sub>2</sub>CO. Spar. sol. MeOH. Stable.

Witt, Noelting, Grandmougin, *Ber.*, 1890, **23**, 3636.

Auwers, Demuth, *Ann.*, 1927, **451**, 298.

See also previous references.

### 7-Nitroindazole.

Cryst. from EtOH. M.p. 186.5–187.5°.

N-Me: see 7-Nitro-methylindazole.

1-Acetyl: needles from 50% AcOH. M.p. 147°. Sol. Me<sub>2</sub>CO, AcOH. Mod. sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether.

2-Acetyl: yellow leaflets from Et<sub>2</sub>O. M.p. 132.5–134°.

1-Benzoyl: needles from MeOH or EtOH. M.p. 132–3°. Sol. Me<sub>2</sub>CO. Mod. sol. AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, Et<sub>2</sub>O.

2-Benzoyl: yellow needles from Me<sub>2</sub>CO. M.p. 185°.

1-o-Nitrobenzoyl: brownish cryst. from toluene–pet. ether. M.p. 173–5°. Sol. Me<sub>2</sub>CO. Stable.

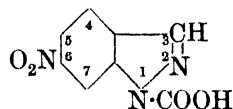
1-p-Nitrobenzoyl: yellow needles from toluene. M.p. 155–7°. Sol. Me<sub>2</sub>CO.

Noelting, *Ber.*, 1904, **37**, 2575.

Auwers, Schwegler, *Ber.*, 1920, **53**, 1211.

Auwers, Demuth, *Ann.*, 1927, **451**, 302.

### 6-Nitroindazole-1-carboxylic Acid



C<sub>8</sub>H<sub>5</sub>O<sub>4</sub>N<sub>3</sub> MW, 207

Me ester: C<sub>9</sub>H<sub>7</sub>O<sub>4</sub>N<sub>3</sub>. MW, 221. Yellow needles from MeOH. M.p. 169°. Sol. AcOH. Spar. sol. EtOH.

Et ester: C<sub>10</sub>H<sub>9</sub>O<sub>4</sub>N<sub>3</sub>. MW, 235. Yellow needles from EtOH. M.p. 147–8°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. MeOH.

Chloride: C<sub>8</sub>H<sub>4</sub>O<sub>3</sub>N<sub>3</sub>Cl. MW, 225.5. Needles from Me<sub>2</sub>CO. M.p. 140–1°. Sol. Me<sub>2</sub>CO. Spar. sol. warm Et<sub>2</sub>O, pet. ether.

Amide: C<sub>8</sub>H<sub>6</sub>O<sub>3</sub>N<sub>4</sub>. MW, 206. Yellow needles from AcOH. M.p. 219–20°. Spar. sol. org. solvents.

Anilide: C<sub>14</sub>H<sub>10</sub>O<sub>3</sub>N<sub>4</sub>. MW, 282. M.p. 205–7°.

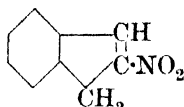
Auwers, Demuth, *Ann.*, 1927, **457**, 297.

### 6-Nitroindazole-3-carboxylic Acid.

Yellow plates from AcOH. M.p. 277–82° decomp.

*Et ester*: needles from EtOH. M.p. 264–6°. Sol. MeOH, EtOH. NaOH  $\rightarrow$  orange-red sol. N-Me:  $C_{11}H_{11}O_4N_3$ . MW, 249. Cryst. from MeOH. M.p. 178°.

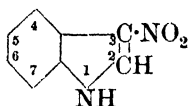
Hahn, Just, *Ber.*, 1932, **65**, 721.

**2-Nitroindene**

$C_9H_7O_2N$  MW, 161

Cryst. from AcOH. M.p. 141°. Zn dust + AcOH  $\rightarrow$  2-hydrindone oxime.

Wallach, Beschke, *Ann.*, 1904, **336**, 2.

**3-Nitroindole**

$C_8H_6O_2N_2$  MW, 162

Yellow needles from  $C_6H_6$ . M.p. 210°.

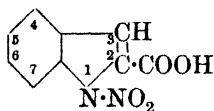
N-Et:  $C_{10}H_{10}O_2N_2$ . MW, 190. Needles from, pet. ether. M.p. 102°.

Angelico, Velardi, *Gazz. chim. ital.*, 1904, **34**, ii, 60.

**6-Nitroindole.**

Yellow prisms from 70% EtOH. M.p. 139–140.5°.

Majima, Kotake, *Ber.*, 1930, **63**, 2241.

**1-Nitroindole-2-carboxylic Acid**

$C_9H_6O_4N_2$  MW, 206

Yellow cryst. from MeOH. M.p. 189° decomp. Spar. sol. usual org. solvents. Sol. alkalis, alk. carbonates, conc.  $H_2SO_4$  with yellowish-red col. Reduces Fehling's on heating.

Reissert, *Ber.*, 1896, **29**, 663.

**3-Nitroindole-2-carboxylic Acid.**

Yellow leaflets from xylene. M.p. 203° decomp. Above m.p.  $\rightarrow$  3-nitroindole.

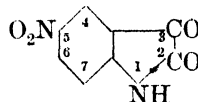
Angelico, Velardi, *Gazz. chim. ital.*, 1904, **34**, ii, 65.

**6-Nitroindole-3-carboxylic Acid.**

Yellow needles from EtOH. Decomp. about 275–8°. Alk. salts spar. sol.  $H_2O$  with orange-red col.

*Et ester*:  $C_{11}H_{10}O_4N_2$ . MW, 234. Yellowish needles from EtOH. M.p. 198–9°.

Majima, Kotake, *Ber.*, 1930, **63**, 2240.

**5-Nitroisatin (5-Nitro- $\psi$ -isatin)**

$C_8H_4O_4N_2$  MW, 192

Yellow needles from EtOH. M.p. 245° decomp. Sol. EtOH. Spar. sol.  $H_2O$ . Sol. alkalis with red col.

N-Acetyl: yellow needles from  $C_6H_6$ . M.p. 193–4°. Insol.  $H_2O$ .

3-Oxime: benzyl ether, yellow needles. M.p. 234–5°. Sol.  $Me_2CO$ ,  $C_6H_6$ . Spar. sol. EtOH.

3-Phenylhydrazone: yellow cryst. from EtOH. M.p. 284°.

N-Me: see 5-Nitro-1-methylisatin.

Rupe, Kersten, *Helv. Chim. Acta*, 1926, **9**, 578.

Liebermann, Kraus, *Ber.*, 1907, **40**, 2501. Korezyński, Marchlewski, *Ber.*, 1902, **35**, 4337.

Schunck, Marchlewski, *Ber.*, 1895, **28**, 546.

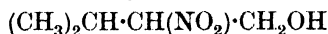
Rupe, Stöcklin, *Helv. Chim. Acta*, 1924, **7**, 564.

**6-Nitroisatin (6-Nitro- $\psi$ -isatin).**

Red needles from EtOH. M.p. 244° decomp.

3-Phenylhydrazone: brown powder from EtOH. M.p. 286°.

See first reference above.

**2-Nitroisoamyl Alcohol (3-Nitro-2-methylbutanol-4, nitroisobutylcarbinol)**

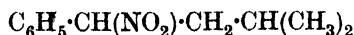
$C_5H_{11}O_3N$  MW, 133

B.p. 138–9°/38 mm.  $D_{20}^{25}$  1.0966. Insol.  $H_2O$ .

Nitrate: b.p. 135°/17 mm. Insol.  $H_2O$ . Spar. volatile in steam.

Shaw, *Chem. Zentr.*, 1898, **1**, 439.

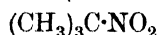
Wieland, Rahn, *Ber.*, 1921, **54**, 1775.

 **$\alpha$ -Nitroisoamylbenzene**

$C_{11}H_{15}O_2N$  MW, 193

B.p. 159–61°/20 mm.  $D_{20}^{25}$  1.08991,  $D_{20}^{20}$  1.07362.  $n_D^{20}$  1.53140.

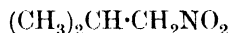
Konowalow, Jegorow, *Chem. Zentr.*, 1899, **1**, 776.

**2-Nitroisobutane** (*tert.*-Nitrobutane) $\text{C}_4\text{H}_9\text{O}_2\text{N}$  MW, 103

Cryst. M.p.  $24^\circ$ . B.p.  $126\text{--}126.5^\circ/748$  mm.  
 Misc. with EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. alkalis.  
 Sn + HCl  $\longrightarrow$  *tert.*-butylamine.

Bewad, *J. prakt. Chem.*, 1893, **48**, 359, 367.

Hass, Hodge, Vanderbilt, U.S.P., 1,976,667, (*Chem. Abstracts*, 1934, **28**, 5830).

**3-Nitroisobutane** $\text{C}_4\text{H}_9\text{O}_2\text{N}$  MW, 103

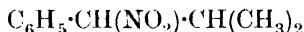
B.p.  $137\text{--}40^\circ$  ( $158\text{--}9^\circ/755$  mm.).  $D^{20}_D$  0.987.

Demole, *Ann.*, 1875, **175**, 142.

**2-Nitroisobutyl Alcohol** $\text{C}_4\text{H}_9\text{O}_3\text{N}$  MW, 119

Needles or plates from MeOH. M.p.  $82^\circ$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

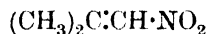
Henry, *Bull. soc. chim.*, 1895, **13**, 1002.

 **$\alpha$ -Nitroisobutylbenzene** $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

Oil. B.p.  $244^\circ$  decomp.,  $145\text{--}6^\circ/25$  mm.  $n^{20}_D$  1.50746.

*Ac-form*: 1-isonitroso-2-methyl-1-phenylpropane. Cryst. M.p. about  $54^\circ$  decomp. Stable only in Et<sub>2</sub>O and in the cold. Sol. Na<sub>2</sub>CO<sub>3</sub>.

Konowalow, *Ber.*, 1895, **28**, 1858; 1896, **29**, 2197.

**1-Nitroisobutylene** $\text{C}_4\text{H}_7\text{O}_2\text{N}$  MW, 101

Yellow oil with pungent odour. B.p.  $154\text{--}8^\circ$  part. decomp.,  $80^\circ/40$  mm.,  $50^\circ/10$  mm.  $D^{20}_D$  1.052. Insol. H<sub>2</sub>O. Sol. alkalis. AlHg in Et<sub>2</sub>O or Zn + AcOH in Et<sub>2</sub>O  $\longrightarrow$  isobutyraldixime. H<sub>2</sub>O at  $100^\circ$   $\longrightarrow$  acetone and nitromethane.

Haitinger, *Ann.*, 1878, **193**, 368, 382.

**1-Nitroisobutyric Acid** $\text{C}_4\text{H}_7\text{O}_4\text{N}$  MW, 133

Cryst. M.p.  $95^\circ$ . Very sol. EtOH, Et<sub>2</sub>O. Sol. hot CHCl<sub>3</sub>. Mod. sol. H<sub>2</sub>O. Very spar.

sol. CS<sub>2</sub>. Decomp. readily, *e.g.*, by heating with H<sub>2</sub>O, xylene or PhNO<sub>2</sub>  $\longrightarrow$  a blue oil.

*Me ester*:  $\text{C}_5\text{H}_9\text{O}_4\text{N}$ . MW, 147. B.p.  $73\text{--}4^\circ/12$  mm.

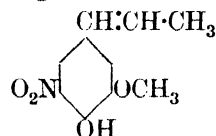
*Amide*:  $\text{C}_4\text{H}_8\text{O}_3\text{N}_2$ . MW, 132. Leaflets from Et<sub>2</sub>O. M.p.  $117\text{--}18^\circ$ . Sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O. Hot dil. H<sub>2</sub>SO<sub>4</sub>  $\longrightarrow$  CO<sub>2</sub>, N<sub>2</sub>O and acetone.

*Nitrile*:  $\text{C}_4\text{H}_5\text{O}_2\text{N}_2$ . MW, 114. Cryst. from ligroin. M.p.  $35^\circ$ . B.p.  $97^\circ/45$  mm.,  $73^\circ/12$  mm. Sol. EtOH. Spar. sol. H<sub>2</sub>O.

Steinkopf, Supan, *Ber.*, 1911, **44**, 2893, 2896.

Piloty, v. Schwerin, *Ber.*, 1901, **34**, 1865.

Pedersen, *J. Phys. Chem.*, 1934, **38**, 559.

**5-Nitroisoeugenol** $\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$  MW, 209

Yellowish-red powder. Decomp. about  $150^\circ$ . Sol. EtOH, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O. Very sol. dil. alkalis.

*Acetyl*: yellowish-brown amorph. powder. Decomp. above  $200^\circ$ . Sol. EtOH, CHCl<sub>3</sub>, AcOH.

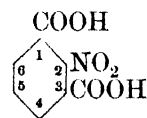
Puxeddu, Cornella, *Gazz. chim. ital.*, 1906, **36**, ii, 451.

**Nitro-isonitroso-ethane.**

*See* Ethylnitrolic Acid.

**Nitro-isonitroso-methane.**

*See* Methylnitrolic Acid.

**2-Nitroisophthalic Acid** $\text{C}_8\text{H}_5\text{O}_6\text{N}$  MW, 211

Prisms from MeOH. M.p.  $315^\circ$  ( $300^\circ$ ). Sol. to 0.216% in H<sub>2</sub>O at  $25^\circ$ . Sol. EtOH, Et<sub>2</sub>O.

*Mono-Me ester*:  $\text{C}_9\text{H}_7\text{O}_6\text{N}$ . MW, 225. Needles from H<sub>2</sub>O. M.p.  $197^\circ$ . Very sol. MeOH. *Chloride*:  $\text{C}_8\text{H}_5\text{O}_5\text{NCl}$ . MW, 243.5. Leaflets from C<sub>6</sub>H<sub>6</sub>. M.p.  $121^\circ$ .

*Di-Me ester*:  $\text{C}_{10}\text{H}_9\text{O}_6\text{N}$ . MW, 239. Needles from H<sub>2</sub>O. M.p.  $135^\circ$ . Insol. pet. ether.

*Monoamide*:  $\text{C}_8\text{H}_6\text{O}_5\text{N}_2$ . MW, 210. Plates from MeOH. M.p.  $252^\circ$ . *Me ester*:  $\text{C}_9\text{H}_8\text{O}_5\text{N}_2$ . MW, 224. Needles from MeOH. M.p.  $190\text{--}1^\circ$ .

Wohl, *Ber.*, 1910, **43**, 3480.

Noelting, Gachot, *Ber.*, 1906, **39**, 73.

**4-Nitroisophthalic Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $258-9^\circ$  ( $245^\circ$ ). Very sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Sol. to 0.967% in  $\text{H}_2\text{O}$  at  $25^\circ$ .  $k$  (first)  $= 1.03 \times 10^{-2}$  at  $25^\circ$ ; (second)  $= 2.63 \times 10^{-4}$  at  $25^\circ$ .

1-*Me ester*:  $\text{C}_9\text{H}_7\text{O}_6\text{N}$ . MW, 225. Needles. from  $\text{C}_6\text{H}_6$ . M.p.  $153.5-154^\circ$ .  $k = 1.09 \times 10^{-2}$  at  $25^\circ$ .

3-*Me ester*: powder from  $\text{C}_6\text{H}_6$ . M.p.  $192-4^\circ$ .  $k = 8.4 \times 10^{-4}$  at  $25^\circ$ .

Di-*Me ester*: yellowish cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $87-88.5^\circ$ .

Axer, *Monatsh.*, 1920, **41**, 159.

Noyes, *Am. Chem. J.*, 1888, **10**, 485.

**5-Nitroisophthalic Acid.**

Leaflets +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd.  $249^\circ$  ( $255-6^\circ$ ). Very sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .

Di-*Me ester*:  $\text{C}_{10}\text{H}_9\text{O}_6\text{N}$ . MW, 239. Needles. M.p.  $123^\circ$  ( $121^\circ$ ). Sol.  $\text{EtOH}$ .

Di-*Et ester*:  $\text{C}_{12}\text{H}_{13}\text{O}_6\text{N}$ . MW, 267. Needles from  $\text{EtOH}$ . M.p.  $83.5^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ , cold  $\text{EtOH}$ .

Möller, *Ber.*, 1909, **42**, 433 (Note).

Huisinga, *Rec. trav. chim.*, 1908, **27**, 265.

Meyer, Wesche, *Ber.*, 1917, **50**, 444.

**1-Nitroisopropyl Alcohol**

$\text{C}_3\text{H}_7\text{O}_3\text{N}$  MW, 105

F.p. about  $-20^\circ$ . B.p.  $112^\circ/13$  mm.  $D_{18}^{18}$  1.1910. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .  $\text{CrO}_3 \rightarrow$  nitroacetone.

Acetyl: b.p.  $94-5^\circ/8$  mm.  $D_4^{20}$  1.1588.  $n_D^{20}$  1.4242.

Henry, *Chem. Zentr.*, 1898, **II**, 887.

Schmidt, Rutz, *Ber.*, 1928, **61**, 2145.

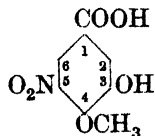
**Nitro-*p*-isopropylbenzoic Acid.**

See Nitrocuminic Acid.

**Nitroisopropylcresol.**

See Nitrocarvacrol and Nitrothymol.

**5-Nitroisovanillic Acid** (5-Nitro-3-hydroxy-anisic acid)



$\text{C}_8\text{H}_7\text{O}_6\text{N}$  MW, 213

Needles from  $\text{H}_2\text{O}$  or  $\text{C}_6\text{H}_6$ . M.p.  $174^\circ$ . Sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .

Acetyl: needles. M.p.  $168-9^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

Matsomoto, *Ber.*, 1878, **11**, 133.

Klemenc, *Monatsh.*, 1914, **35**, 95.

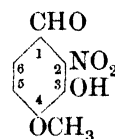
**6-Nitroisovanillic Acid** (6-Nitro-3-hydroxy-anisic acid).

Yellow prisms from boiling  $\text{H}_2\text{O}$ . M.p.  $181^\circ$  decomp. Sol.  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{AcOH}$ , boiling  $\text{H}_2\text{O}$ . Spar. sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Alc.  $\text{FeCl}_3 \rightarrow$  greenish-brown col. Alk. sols are orange-yellow.

Me ester:  $\text{C}_9\text{H}_9\text{O}_6\text{N}$ . MW, 227. Cryst. from  $\text{EtOH.Aq}$ . M.p.  $143^\circ$ .

Acetyl: plates from  $\text{EtOH.Aq}$ . M.p.  $214^\circ$  decomp.

Greenwood, Robinson, *J. Chem. Soc.*, 1932, 1371.

**2-Nitroisovanillin**

$\text{C}_8\text{H}_7\text{O}_5\text{N}$

MW, 197

Leaflets from  $\text{EtOH}$ . M.p.  $148-9^\circ$ . Turns brown in sunlight.

Me ether: see 2-Nitroveratric Aldehyde.

Et ether:  $\text{C}_{10}\text{H}_{11}\text{O}_5\text{N}$ . MW, 225. Cryst. from  $\text{EtOH}$ . M.p.  $76-7^\circ$ .

Phenylhydrazon: dark violet needles from  $\text{AcOH}$ . M.p.  $157-8^\circ$ .

Pschorr, Stoehr, *Ber.*, 1902, **35**, 4396.

Späth, Tharrer, *Ber.*, 1933, **66**, 912.

**5-Nitroisovanillin.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $113^\circ$ . Sol. most org. solvents. Mod. sol.  $\text{H}_2\text{O}$ .

Acetyl: yellowish needles from  $\text{H}_2\text{O}$ . M.p.  $86^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{H}_2\text{O}$ . Phenylhydrazon: yellow needles from  $\text{EtOH}$ . M.p.  $165^\circ$ .

Me ether: see 5-Nitroveratric Aldehyde.

Benzoyl: needles from  $\text{EtOH}$ . M.p.  $120-1^\circ$ . Phenylhydrazon: orange cryst. from  $\text{EtOH}$ . M.p.  $205-6^\circ$ .

Phenylhydrazon: yellowish-red prisms from  $\text{EtOH}$ . M.p.  $170^\circ$ .

Pschorr, Stoehr, *Ber.*, 1902, **35**, 4398.

Hinkel, Ayling, Morgan, *J. Chem. Soc.*, 1935, 817.

**6-Nitroisovanillin.**

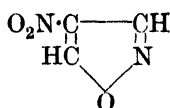
Yellow needles from  $\text{EtOH}$ . M.p.  $189^\circ$ . Sol.  $\text{AcOH}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , hot  $\text{EtOH}$ . Spar. sol. ligroin, hot  $\text{H}_2\text{O}$ . Darkens rapidly in light.

Phenylhydrazon: red leaflets from  $\text{EtOH}$ . M.p.  $200-1^\circ$ .

Me ether: see 6-Nitroveratric Aldehyde.

Pschorr, Stoehr, *Ber.*, 1902, **35**, 4394.

## 4-Nitroisoxazole



$C_3H_2O_3N_2$  MW, 114

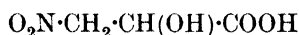
Plates from  $Et_2O$ -ligroin. M.p. 46–7°. Sol. EtOH,  $Et_2O$ . Spar. sol.  $CS_2$ . Very spar. sol. ligroin. Sol. alkalis with deep yellow col.

Hill, Lorrey, *Am. Chem. J.*, 1899, **22**, 106.

## 7-Nitrokairolin.

See under 7-Nitro-1 : 2 : 3 : 4-tetrahydroquinoline.

## Nitrolactic Acid

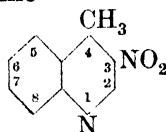


$C_3H_5O_5N$  MW, 135

Prisms from  $Et_2O$ - $CHCl_3$ . M.p. 76–7°. Very sol.  $H_2O$ , EtOH,  $Et_2O$ . Spar. sol.  $CHCl_3$ .  $Zn + HCl \rightarrow$  isoserine.

Hill, Black, *Am. Chem. J.*, 1904, **32**, 231.

## 3-Nitrolepidine



$C_{10}H_8O_2N_2$  MW, 188

Prisms from hot  $H_2O$ . M.p. 118°. Volatile in steam.

Badische, D.R.P., 335,197, (*Chem. Abstracts*, 1923, **17**, 1803).

## 8-Nitrolepidine.

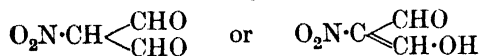
Leaflets from EtOH. M.p. 126–7°. Mod. sol. cold EtOH.

Busch, Koenigs, *Ber.*, 1890, **23**, 2687.

## Nitromalic Acid.

See under Malic Acid.

## Nitromalondialdehyde



$C_3H_3O_4N$  MW, 117

Prisms from  $Et_2O$ . M.p. 50–1°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. ligroin. Decomp. in aq. sol.  $\rightarrow$  formic acid and trinitrobenzene. Na deriv. with hydrazine sulphate  $\rightarrow$  a red ppt. which with warm HCl  $\rightarrow$  4-nitropyrazole. With acetone in alk. sol.  $\rightarrow$  p-nitrophenol.

*Dioxime*: known only in form of aq. sol. of Na salt. Slight excess of HCl + aq. sol. of Na salt  $\rightarrow$  yellow ppt.  $\rightarrow$  nitroisoxazole.

*Monoanil*: yellow plates from EtOH. M.p. 143–4°. *Oxime*: yellow cryst. M.p. 162°. *Oxime acetate*: m.p. 115–16°.

*Dianil*: yellow needles from EtOH.Aq. M.p. 93–4°.

*Mono-p-tolylimide*: m.p. 176–7°.

*Di-p-tolylimide*: m.p. 138°.

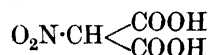
*Monophenylhydrazone*: yellow needles. M.p. 101°, re-solidifies on further heating  $\rightarrow$  4-nitro-1-phenylpyrazole.

*Di-phenylhydrazone*: orange-red solid. M.p. 98° decomp. Heat with  $C_2H_5OH \rightarrow$  4-nitro-1-phenylpyrazole.

Hill, Torrey, *Am. Chem. J.*, 1899, **22**, 89.

Hill, Sanger, *Ber.*, 1882, **15**, 1908.

## Nitromalonic Acid



$C_3H_3O_6N$  MW, 149

*Di-Me ester*:  $C_5H_7O_6N$ . MW, 177. Yellowish oil. Readily sol. alkalis.  $NH_4$  salt: m.p. about 166° decomp. *K salt*: yellow plates from EtOH. M.p. 206–206.5°. Very sol.  $H_2O$ .

*Di-Et ester*:  $C_7H_{11}O_6N$ . MW, 205. Colourless oil with fruity odour. B.p. 152–3°/37–38 mm., 134–5°/14 mm., 127°/10 mm.  $D_4^{20}$  1.220,  $D_4^{20}$  1.1988. Insol.  $H_2O$ .  $NH_3$ .Aq. at 100°  $\rightarrow$  nitroacetamide.  $NH_4$  salt: leaflets from EtOH. M.p. 152–3° (162°). Very sol.  $H_2O$ . Insol.  $Et_2O$ . *K salt*: prisms from EtOH. M.p. 154°. Very sol.  $H_2O$ , EtOH, hot  $Me_2CO$ . Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , ligroin.

*Diamide*:  $C_3H_5O_4N_3$ . MW, 147. Prisms from  $H_2O$ . M.p. 172°. Insol. EtOH.

*Mononitrile*: see Nitrocyanoacetic Acid.

*Dianilide*:  $C_{15}H_{13}O_4N_3$ . MW, 299. Pale yellow plates from EtOH. M.p. 141–2°.

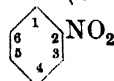
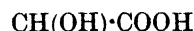
*Di-N-methylanilide*:  $C_{17}H_{17}O_4N_3$ . MW, 327. Prisms. M.p. 156° decomp.

Ulpiani, *Gazz. chim. ital.*, 1912, **42**, i, 223, 400.

Franchimont, Klobbie, *Rec. trav. chim.*, 1889, **8**, 283.

Ratz, *Monatsh.*, 1904, **25**, 60.

## o-Nitromandelic Acid



$C_8H_7O_5N$  MW, 197

d-.

Plates from  $CHCl_3$ . M.p. 100–1°.  $[\alpha]_{D}^{20}$  + 490° in  $Me_2CO$ .



MW, 212

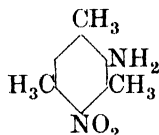
Yellow needles from AcOH. M.p. 255–7°.

Teppema, Sebrell, *J. Am. Chem. Soc.*, 1927, **49**, 1780.

Naugatuck, F.P., 741,910, (*Chem. Abstracts*, 1933, **27**, 3642).

Sebrell, U.S.P., 1,958,770, (*Chem. Abstracts*, 1934, **28**, 4632).

**4-Nitromesidine** (4-Nitro-2-aminomesitylene)



$C_9H_{12}O_2N_2$  MW, 180

Golden-yellow needles from EtOH. M.p. 75°. Very sol. EtOH, Et<sub>2</sub>O.

Acetyl: needles from EtOH. M.p. 191°. Mod. sol. boiling EtOH. Sol. conc. HCl.

Benzoyl: cryst. M.p. 168.5°.

m-Nitrobenzoyl: m.p. 207–8°.

Benzenesulphonyl: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 162–3°.

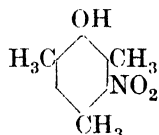
Morgan, Micklethwait, *J. Chem. Soc.*, 1906, **89**, 1299.

Hübner, v. Schack, *Ber.*, 1877, **10**, 1711.

Ladenburg, *Ann.*, 1875, **179**, 165.

Kuster, Stallberg, *Ann.*, 1894, **278**, 214.

**Nitromesitol** (4-Nitro-2-hydroxymesitylene, 3-nitro-2 : 4 : 6-trimethylphenol)

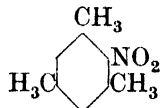


$C_9H_{11}O_3N$  MW, 181

Yellow leaflets from H<sub>2</sub>O. M.p. 64°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O. Volatile in steam.

Knecht, *Ann.*, 1882, **215**, 98.

**Nitromesitylene**



$C_9H_{11}O_2N$  MW, 165

Prisms from EtOH. M.p. 44° (41–2°). B.p. 255°. Very sol. hot EtOH. CrO<sub>3</sub> in AcOH at 60–70° → 4-nitro-3 : 5-dimethylbenzoic acid.

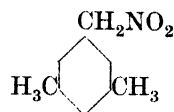
Bamberger, Rising, *Ber.*, 1900, **33**, 3625.

Ladenburg, *Ann.*, 1875, **179**, 170.

Francis, *Ber.*, 1906, **39**, 3801.

Powell, Johnson, *Organic Syntheses*, 1934, **XIV**, 68.

**ω-Nitromesitylene** (3 : 5-Dimethylphenyl-nitromethane)



$C_9H_{11}O_2N$  MW, 165

Cryst. from EtOH. M.p. 46.8°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether. Volatile in steam. Heat of comb. C<sub>p</sub> 1206.33 Cal. Sol. caustic alkalis and hot Na<sub>2</sub>CO<sub>3</sub>. Aq. giving Na salt of aci-form.

Aci-Form: ω-isonitromesitylene; 3 : 5-dimethylphenylisonitromethane

(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>·CH·NO<sub>2</sub>H

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. about 63° decomp. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOEt, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether. Passes into normal nitro form on standing, especially in sunlight.

Konowalow, *Ber.*, 1895, **28**, 1862; 1896, **29**, 2201.

**1-Nitromesityl oxide** (5-Nitro-2-methyl-2-pentenone-4)

(CH<sub>3</sub>)<sub>2</sub>C·CH·CO·CH<sub>2</sub>NO<sub>2</sub>

$C_8H_9O_3N$  MW, 143

Yellow pungent liq. B.p. 95–6°/23 mm. D<sub>27.3</sub><sup>20</sup> 1.212. Semicarbazide → nitroacetone semicarbazone.

Anil: yellow leaflets. M.p. 84–5°.

Harries, *Ann.*, 1901, **319**, 248.

**Nitromethane**

CH<sub>3</sub>·NO<sub>2</sub>

CH<sub>3</sub>O<sub>2</sub>N MW, 61

F.p. – 17° (– 28.5°). B.p. 101–101.5°/764.7 mm. D<sub>4</sub><sup>4</sup> 1.1580, D<sub>15</sub><sup>15</sup> 1.1441, D<sub>20</sub><sup>20</sup> 1.1382, D<sub>4</sub><sup>4</sup> 1.1437, D<sub>4</sub><sup>25</sup> 1.1297. n<sub>D</sub><sup>21.6</sup> 1.38133, n<sub>D</sub><sup>25</sup> 1.37970. Heat of comb. C<sub>p</sub> 169.8 Cal. (180.90 Cal.) (vapour), 169.5 Cal. (liq.); C<sub>v</sub> 170.25 Cal. (vapour), 169.95 Cal. (liq.). Aq. sol. reacts acid to litmus. SnCl<sub>2</sub> → methylamine and N-methylhydroxylamine. Nascent HNO<sub>2</sub> → methylnitrolic acid. Hot conc. H<sub>2</sub>SO<sub>4</sub> → CO and hydroxylamine.

Steinkopf, Kirchhoff, *Ber.*, 1909, **42**, 3439.

Wahl, *Bull. soc. chim.*, 1909, **5**, 180.

Walden, *Ber.*, 1907, **40**, 3216, 4301.

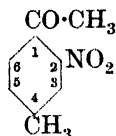
Hirano, *Chem. Abstracts*, 1931, **25**, 69.

Wang, Tseng, *ibid.*, 681.

Pritzl, Adkins, *J. Am. Chem. Soc.*, 1931, **53**, 234.

Krause, Swiss P., 74,333, (*Chem. Abstracts*, 1917, **11**, 2027).

Whitmore, Whitmore, *Organic Syntheses*, Collective Vol. I, 393.

**2-Nitro-*p*-methylacetophenone** (*Methyl 2-nitro-*p*-tolyl ketone*) $C_9H_9O_3N$ 

MW, 179

Yellow oil. Zn dust + NaOH  $\rightarrow$  6 : 6'-di-methylindigo.

Duff, *J. Chem. Soc.*, 1914, 105, 2185.

**3-Nitro-*p*-methylacetophenone** (*Methyl 3-nitro-*p*-tolyl ketone*).

Yellow needles from pet. ether. M.p. 61°. Sol. EtOH. Spar. sol. cold pet. ether. Dil.  $HNO_3 \rightarrow$  2-nitro-*p*-toluic acid.

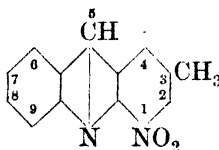
Oxime : prisms from EtOH. M.p. 133°.

Semicarbazone : yellow powder from AcOH. M.p. 262° decomp.

2 : 4-Dinitrophenylhydrazone : orange-yellow plates from xylene. M.p. 232°.

Errera, *Gazz. chim. ital.*, 1891, 21, i, 92.

Brady, Day, *J. Chem. Soc.*, 1934, 120.

**1-Nitro-3-methylacridine** $C_{14}H_{10}O_2N_2$ 

MW, 238

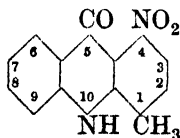
Brown needles from MeOH. M.p. 201-2°.

Mayer, Stein, *Ber.*, 1917, 50, 1317.

**3-Nitro-5-methylacridine.**

Brownish-yellow needles from EtOH. Does not melt below 300°.

Jensen, Rethwisch, *J. Am. Chem. Soc.*, 1928, 50, 1149.

**4-Nitro-1-methylacridone** $C_{14}H_{10}O_3N_2$ 

MW, 254

Yellow prisms from  $C_6H_6$ . Does not melt below 300°.

Clemons, Perkin, Robinson, *J. Chem. Soc.*, 1924, 125, 1774.

**8-Nitro-2-methylacridone.**

Light brown cryst. from EtOH.Aq. Does not melt below 300°. Mod. sol.  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  greenish-blue col. Hot alkalis  $\rightarrow$  reddish-violet fluor.

Tanasescu, Macarovici, *Bull. soc. chim.*, 1933, 53, 372.

**1-Nitro-3-methylacridone.**

Red cryst. from  $C_6H_6$ . M.p. 250°.

Mayer, Stein, *Ber.*, 1917, 50, 1317.

**1-Nitro-10-methylacridone** (*1-Nitro-N-methylacridone*).

Yellow needles from EtOH. M.p. 168°.

Lehmstedt, Hundertmark, *Ber.*, 1931, 64, 2391.

**3-Nitro-10-methylacridone** (*3-Nitro-N-methylacridone*).

Yellow needles from AcOH. M.p. 276°. Mod. sol. usual org. solvents.

Lehmstedt, Hundertmark, *Ber.*, 1931, 64, 2390.

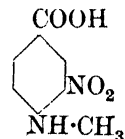
**9-Nitro-10-methylacridone** (*9-Nitro-N-methylacridone*).

Yellow prisms from xylene. M.p. 176-7°.

Burton, Gibson, *J. Chem. Soc.*, 1924, 125, 2503.

**N-Nitromethylamine.**

See Methylnitramine.

**3-Nitro-*p*-methylaminobenzoic Acid** $C_8H_8O_4N_2$ 

MW, 196

Yellow needles from EtOH. M.p. 303-5° (288°).

*Me ester* :  $C_9H_{10}O_4N_2$ . MW, 210. Yellow cryst. from  $C_6H_6$ -ligroin. M.p. 145°. Sol. warm EtOH,  $C_6H_6$ , AcOH. Very spar. sol. ligroin.

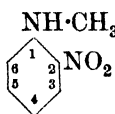
*Et ester* :  $C_{10}H_{12}O_4N_2$ . MW, 224. Yellow needles from EtOH. M.p. 101-2°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH, conc. HCl. Insol.  $H_2O$ .

*N-Acetyl* : yellow needles from EtOH. M.p. 190°.

Noelting, Demant, *Ber.*, 1904, 37, 1029.

Thieme, *J. prakt. Chem.*, 1891, 43, 458.

Reverdin, de Luc, *Bull. soc. chim.*, 1908, 3, 133.

**o-Nitro-N-methylaniline** $C_7H_8O_2N_2$ 

MW, 152

Red needles with blue reflex from pet. ether. M.p. 37°. Sol. usual org. solvents. Spar. sol. cold  $H_2O$ , ligroin. Sol. conc. acids.

N-Acetyl: m.p. 70°.

N-Nitroso: yellowish needles from EtOH. M.p. 36°.

Fischer, Veiel, *Ber.*, 1905, **38**, 321.

Hempel, *J. prakt. Chem.*, 1890, **41**, 168.

**m-Nitro-N-methylaniline.**

Reddish-yellow needles or prisms from EtOH. M.p. 68°. Sol. EtOH,  $Et_2O$ . Mod. sol. hot  $H_2O$ .

N-Acetyl: needles from  $H_2O$ . M.p. 94–5°.

N-Benzoyl: m.p. 104–5°.

N-Nitroso: leaflets from EtOH or  $Me_2CO$ . M.p. 76° (68–70°). Sol. EtOH. Volatile in steam.

Ullmann, *Ann.*, 1903, **327**, 112.

Meldola, Salmon, *J. Chem. Soc.*, 1888, **53**, 777.

Hodgson, Smith, *J. Chem. Soc.*, 1931, 1509.

Schmidt, Schumacher, *Ber.*, 1921, **54**, 1419.

**p-Nitro-N-methylaniline.**

Brownish-yellow prisms with violet reflex from EtOH. M.p. 152°. Sol. EtOH,  $C_6H_6$ . Spar. sol. ligroin.

N-Formyl: yellow prisms from EtOH. M.p. 118–20°.

N-Acetyl: plates from  $H_2O$ . M.p. 152–3°.

N-Benzoyl: prisms from EtOH.Aq. M.p. 111–12°.

N-β-Chloroethyl: greenish-brown cryst. from EtOH. M.p. 90°.

N-Benzyl: yellow needles from MeOH. M.p. 68–9°.

N-Nitroso: needles from hot EtOH. M.p. 104° (101°). Sol. usual solvents. Spar. sol.  $H_2O$ , ligroin.

Meldola, Salmon, *J. Chem. Soc.*, 1888, **53**, 775.

Morgan, Grist, *J. Chem. Soc.*, 1918, **113**, 690.

Hodgson, Smith, *J. Chem. Soc.*, 1931, 1510.

Stoermer, Hoffmann, *Ber.*, 1898, **31**, 2528.

**N-Nitro-N-methylaniline.**

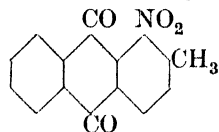
See Methylphenylnitramine.

**Nitro-N-methylanisidine.**

See under Nitroanisidine.

**Nitro-N-methylanthranilic Acid.**

See under Nitroanthranilic Acid.

**1-Nitro-2-methylanthraquinone** $C_{15}H_8O_4N$ 

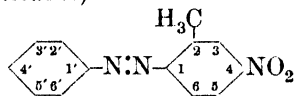
MW, 267

Pale yellow needles from AcOH. M.p. 269–70°. Sol.  $PhNO_2$ . Spar. sol.  $C_6H_6$ ,  $CHCl_3$ , AcOH, AcOEt. Almost insol. EtOH,  $Et_2O$ .  $Na_2S.Aq.$  → 1-amino-2-methylanthraquinone. Conc.  $H_2SO_4$  → yellow sol. → reddish-brown → brown on heating. Sol. gives alkali-soluble, purple ppt. on dilution.

Römer, Link, *Ber.*, 1883, **16**, 697.

Fierz, U.S.P., 1,540,467, (*Chem. Abstracts*, 1925, **19**, 2210).

Locker, Fierz, *Helv. Chim. Acta*, 1927, **10**, 642.

**4-Nitro-2-methylazobenzene** (6-Benzene-azo-m-nitrotoluene) $C_{13}H_{11}O_2N_3$ 

MW, 241

Red prisms from EtOH, red needles from  $H_2O$ . M.p. 98–9°. Sol. usual solvents. Spar. sol.  $H_2O$ , pet. ether.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1174.

**6-Nitro-2-methylazobenzene** (2-Benzene-azo-m-nitrotoluene).

Red oil. B.p. 215°/11 mm.

See previous reference.

**2'-Nitro-2-methylazobenzene** (2-o-Nitro-benzeneazotoluene).

Brownish-red needles from EtOH. M.p. 108–9°. Sol.  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ , AcOH, hot EtOH, hot ligroin.

Bamberger, Hübner, *Ber.*, 1903, **36**, 3818.

**2-Nitro-4-methylazobenzene** (4-Benzene-azo-m-nitrotoluene).

Red leaflets or needles. M.p. 71–71.5°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ , AcOH, hot EtOH, hot ligroin.

Bamberger, Hübner, *Ber.*, 1903, **36**, 3821.

### 3-Nitro-4-methylazobenzene

**3-Nitro-4-methylazobenzene** (4-Benzene-azo-o-nitrotoluene).

Orange-red or golden needles from EtOH. M.p. 105°.

Meisenheimer, *Ber.*, 1920, **53**, 367.

Burns, McCombie, Scarborough, *J. Chem. Soc.*, 1928, 2931.

**2'-Nitro-4-methylazobenzene** (4-o-Nitrobenzeneazotoluene).

Orange-red needles from EtOH. M.p. 88°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, Me<sub>2</sub>CO, AcOH, C<sub>6</sub>H<sub>6</sub>, hot EtOH, hot ligroin.

Bamberger, Hübner, *Ber.*, 1903, **36**, 3819.

**4'-Nitro-4-methylazobenzene** (4-p-Nitrobenzeneazotoluene).

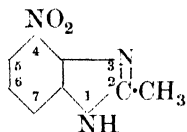
Red needles from EtOH. M.p. 183° (180°). Sol. CHCl<sub>3</sub>, hot ligroin. Spar. sol. EtOH, Et<sub>2</sub>O.

Bamberger, *Ber.*, 1902, **35**, 1427.

Burns, McCombie, Scarborough, *J. Chem. Soc.*, 1928, 2931.

Bigiavi, Sabatelli, *Gazz. chim. ital.*, 1927, **57**, 557.

### 4-Nitro-2-methylbenziminazole



C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub> MW, 177.

Yellowish needles from EtOH. Aq. M.p. 217°

Borsche, Rantschew, *Ann.*, 1911, **379**, 164.

### 5-Nitro-2-methylbenziminazole.

Yellowish needles or plates from H<sub>2</sub>O. M.p. 221°. Sol. hot H<sub>2</sub>O, min. acids, caustic alkalis.

Kym, Ratner, *Ber.*, 1912, **45**, 3245.

Kym, Jurkowski, *Ber.*, 1916, **49**, 2689.

Phillips, *J. Chem. Soc.*, 1928, 176.

### 6-Nitro-2-methylbenziminazole.

Yellowish-brown needles from H<sub>2</sub>O. M.p. 219° (216°). Sol. hot H<sub>2</sub>O, EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, toluene. Mod. sol. Et<sub>2</sub>O. Sol. dil. acids.

Heim, *Ber.*, 1888, **21**, 2307.

Fischer, Hess, *Ber.*, 1903, **36**, 3970.

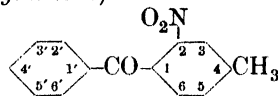
### 6-Nitro-3-methylbenziminazole.

Needles from hot H<sub>2</sub>O. M.p. 199-200°. Sol. EtOH. Spar. sol. cold H<sub>2</sub>O. Very sol. dil. HCl. Sol. dil. NaOH with yellow col.

Kym, Ringer, *Ber.*, 1915, **48**, 1676.

### 188 Nitromethylbenzophenone-carboxylic Acid

**2-Nitro-4-methylbenzophenone** (Phenyl 2-nitro-p-tolyl ketone)



C<sub>14</sub>H<sub>11</sub>O<sub>3</sub>N MW, 241

Plates or needles from EtOH. M.p. 126-7°. Sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, hot AcOH. Sublimes easily. CrO<sub>3</sub> in AcOH → benzoic acid + p-nitrobenzoic acid.

Plascuda, Zinke, *Ber.*, 1874, **7**, 983.

See also Limpricht, *Ann.*, 1895, **286**, 324.

**3-Nitro-4-methylbenzophenone** (Phenyl 3-nitro-p-tolyl ketone).

Cryst. from EtOH or AcOH in pale yellow plates. M.p. 130-2°. Very sol. C<sub>6</sub>H<sub>6</sub>, Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOH, CS<sub>2</sub>. Sol. EtOH, Et<sub>2</sub>O, ligroin. Spar. sol. H<sub>2</sub>O.

Blakey, Scarborough, *J. Chem. Soc.*, 1928, 2492.

Weiss, Katz, *Monatsh.*, 1928, **50**, 109.

Chardonnens, *Helv. Chim. Acta*, 1929, **12**, 654.

**2'-Nitro-4-methylbenzophenone** (2-Nitrophenyl p-tolyl ketone).

Prisms from EtOH or AcOH. M.p. 155°. Sol. warm C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOH. Spar. sol. hot EtOH, Et<sub>2</sub>O, ligroin.

Kliegl, *Ber.*, 1908, **41**, 1845.

Boëtius, Römisch, *Ber.*, 1935, **68**, 1931.

**3'-Nitro-4-methylbenzophenone** (3-Nitrophenyl p-tolyl ketone).

Leaflets. M.p. 111°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH. Can be distilled in small quantities.

Limpricht, Lenz, *Ann.*, 1895, **286**, 307.

**4'-Nitro-4-methylbenzophenone** (4-Nitrophenyl p-tolyl ketone).

Needles. M.p. 122-4°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOH, CS<sub>2</sub>. Sublimes.

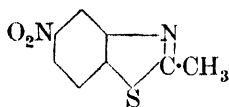
*Oxime*: needles from Et<sub>2</sub>O-ligroin. M.p. 145°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Less sol. ligroin.

*Phenylhydrazone*: red needles from EtOH. M.p. 154°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. ligroin.

Limpricht, Samietz, *Ann.*, 1895, **286**, 321.

**Nitromethylbenzophenone-carboxylic Acid.**

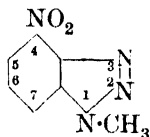
See Nitrotoluybenzoic Acid.

**5-Nitro-2-methylbenzthiazole** (4-Nitro-1-methylbenzthiazole) $C_8H_6O_2N_2S$ 

MW, 194

Long needles from EtOH,  $C_6H_6$ , or AcOH. M.p.  $139^\circ$ . Stable to alkalis. Sol. conc.  $H_2SO_4$ .

Fries, *Ann.*, 1927, **454**, 177.

**4-Nitro-1-methyl-1 : 2 : 3-benztriazole** $C_7H_6O_2N_4$ 

MW, 178

Needles from EtOH. M.p.  $173^\circ$ .

Fries, Güterbock, Kühn, *Ann.*, 1934, **511**, 232.

**5-Nitro-1-methyl-1 : 2 : 3-benztriazole.**

Cryst. from EtOH. M.p.  $163^\circ$ . Sol. hot  $C_6H_6$ ,  $CHCl_3$ .

Pinnow, Koch, *Ber.*, 1897, **30**, 2852.

**6-Nitro-1-methyl-1 : 2 : 3-benztriazole.**

Pale yellow needles from EtOH. M.p.  $187^\circ$ .

Brady, Reynolds, *J. Chem. Soc.*, 1930, 2672.

**7-Nitro-1-methyl-1 : 2 : 3-benztriazole.**

Pale yellow needles from EtOH. M.p.  $203^\circ$ .

Fries, Güterbock, Kühn, *Ann.*, 1934, **511**, 232.

**7-Nitro-5-methyl-1 : 2 : 3-benztriazole.**

Pale yellow needles from EtOH. M.p.  $277^\circ$  decomp.

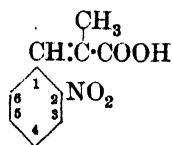
Lindemann, Krause, *J. prakt. Chem.*, 1927, **115**, 256.

**Nitro-*N*-methylbenzylamine.**

See under Nitrobenzylamine.

**Nitromethylcarbostyryl.**

See Nitro-2-hydroxymethylquinoline.

**2-Nitro- $\alpha$ -methylcinnamic Acid** $C_{10}H_9O_4N$ 

MW, 207

Cryst. from EtOH.Aq. M.p.  $164-5^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ , ligroin.  $KMnO_4 \rightarrow o$ -nitrobenzoic acid.

Edeleano, *Ber.*, 1887, **20**, 620.

**3-Nitro- $\alpha$ -methylcinnamic Acid.**

Powder. M.p.  $197.5^\circ$ . Sol. warm EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ . Spar. sol. ligroin.

*Et ester*:  $C_{12}H_{13}O_4N$ . MW, 235. B.p.  $197^\circ/11$  mm.

v. Miller, Rohde, *Ber.*, 1890, **23**, 1900.

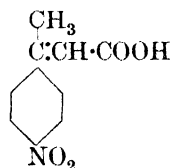
Maxwell, Adams, *J. Am. Chem. Soc.*, 1930, **52**, 2967.

**4-Nitro- $\alpha$ -methylcinnamic Acid.**

Rhombic cryst. from AcOH. M.p.  $208^\circ$ . Mod. sol. hot EtOH, AcOH. Spar. sol. other solvents.  $KMnO_4 \rightarrow p$ -nitrobenzoic acid.

*Me ester*:  $C_{11}H_{11}O_4N$ . MW, 221. Leaflets from EtOH. M.p.  $115^\circ$ .

Edeleano, *Ber.*, 1887, **20**, 620.

**4-Nitro- $\beta$ -methylcinnamic Acid** $C_{10}H_9O_4N$ 

MW, 207

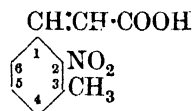
Pale yellowish needles from AcOH. M.p.  $168-9^\circ$ . Dil.  $HNO_3$  at  $160^\circ \rightarrow p$ -nitrobenzoic acid.

*Me ester*:  $C_{11}H_{11}O_4N$ . MW, 221. M.p.  $121-2^\circ$ .

*Et ester*:  $C_{12}H_{13}O_4N$ . MW, 235. Cryst. from EtOH. M.p.  $74^\circ$ .

Schroeter, Wulff, *Ber.*, 1907, **40**, 1594.

Vorländer, *ibid.*, 4535.

**2-Nitro-*m*-methylcinnamic Acid** $C_{10}H_9O_4N$ 

MW, 207

Prisms. M.p.  $244^\circ$ .

*Me ester*:  $C_{11}H_{11}O_4N$ . MW, 221. M.p.  $83^\circ$ .

Chakravarti, Venkatasubham, *Chem. Zentr.*, 1934, **I**, 1329.

**4-Nitro-*m*-methylcinnamic Acid.**

Needles from EtOH. M.p.  $251^\circ$ .

*Me ester*: yellow needles from EtOH. M.p. 124°.

See previous reference.

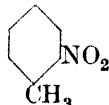
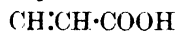
### 6-Nitro-*m*-methylcinnamic Acid.

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 224°.

*Me ester*: prisms from MeOH. M.p. 91°.

See previous reference.

### 3-Nitro-*p*-methylcinnamic Acid



C<sub>10</sub>H<sub>9</sub>O<sub>4</sub>N

MW, 207

Yellow plates or needles. M.p. 173-5° (170-1°). Sol. EtOH, hot H<sub>2</sub>O, Et<sub>2</sub>O. Insol. ligroin.

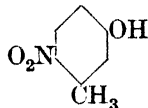
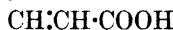
*Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 221. Needles from Et<sub>2</sub>O-ligroin. M.p. 109°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Et ester*: C<sub>12</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 235. Yellow plates from EtOH or Et<sub>2</sub>O-ligroin. M.p. 97°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Gattermann, *Ann.*, 1906, 347, 360.

Hantzik, Bianchi, *Ber.*, 1899, 32, 2285.

### 5-Nitro-4-methyl-*o*-coumaric Acid



C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N

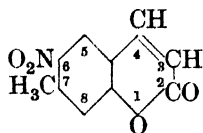
MW, 223

Needles. M.p. 219° decomp.

*Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Needles from EtOH.Aq. M.p. 187°. *Me ether*: C<sub>12</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 251. Needles. M.p. 136°.

Dey, Row, *J. Chem. Soc.*, 1924, 125, 563.

### 6-Nitro-7-methylcoumarin



C<sub>10</sub>H<sub>7</sub>O<sub>4</sub>N

MW, 205

Needles from AcOH. M.p. 223-4°. P<sub>2</sub>S<sub>5</sub> in xylene  $\rightarrow$  6-nitro-7-methylthiocoumarin. Alk. KMnO<sub>4</sub>  $\rightarrow$  5-nitro-4-methylsalicylic acid.

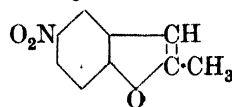
Clayton, *J. Chem. Soc.*, 1910, 97, 1397.

### 8-Nitro-7-methylcoumarin.

Needles from AcOH. M.p. 165-6°.

See previous reference.

### 5-Nitro-2-methylcoumarone



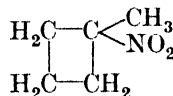
C<sub>9</sub>H<sub>7</sub>O<sub>3</sub>N

MW, 177

Needles from EtOH.Aq. M.p. 97°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, AcOH, AcOEt, ligroin, hot EtOH. Mod. sol. hot H<sub>2</sub>O. Volatile in steam. Warm conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  deep red sol.

Hale, *Ber.*, 1912, 45, 1601.

### 1-Nitro-1-methylcyclobutane



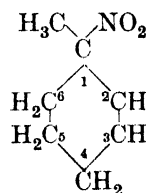
C<sub>5</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 115

B.p. 80-2°/30 mm. D<sub>4</sub><sup>20</sup> 1.0795. n<sub>D</sub><sup>20</sup> 1.4589.

Rosanow, *Chem. Zentr.*, 1916, I, 925.

### 1-Nitro-1-methylcyclohexane



C<sub>7</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 143

M.p. - 71°. B.p. 118-20°/50 mm., 109-10°/40 mm. D<sub>4</sub><sup>20</sup> 1.0547, D<sub>4</sub><sup>20</sup> 1.0384. n<sub>D</sub><sup>20</sup> 1.4598. HNO<sub>3</sub> (D 1.2)  $\rightarrow$  succinic acid and a little oxalic acid.

Nametkin, *Chem. Zentr.*, 1910, II, 1377.

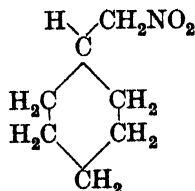
Rosanow, *Chem. Zentr.*, 1924, I, 2426.

**3-Nitro-1-methylcyclohexane** (3-Nitro-hexahydrotoluene).

B.p. 119-20°/40 mm. D<sub>4</sub><sup>20</sup> 1.0547, D<sub>4</sub><sup>20</sup> 1.0382. n<sub>D</sub><sup>20</sup> 1.4618. Alk. KMnO<sub>4</sub>  $\rightarrow$  3-methylcyclohexanone and 1- and 2-methyladipic acids.

Nametkin, *Chem. Zentr.*, 1910, II, 1377.

**$\alpha$ -Nitromethylcyclohexane** ( $\alpha$ -Nitrohexahydrotoluene, cyclohexylnitromethane)



C<sub>7</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 143

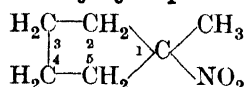
B.p. 123-4°/40 mm., 98°/10 mm.  $D_4^{20}$  1.0482.  $n_D^{20}$  1.4705.  $\text{HNO}_3$  (D 1.2)  $\rightarrow$  adipic acid. Mod. sol. alkalis with yellow col.

Rosanow, *Chem. Zentr.*, 1924, I, 2426.

Zelinsky, *Ber.*, 1908, 41, 2678.

See also previous reference.

### 1-Nitro-1-methylcyclopentane



$\text{C}_6\text{H}_{11}\text{O}_2\text{N}$  MW, 129

Liq. with terpene-like odour. B.p. 177-84°/750 mm. decomp., 92°/40 mm., 86-8°/30 mm.  $D_4^0$  1.0568,  $D_4^{20}$  1.0395.  $n_D^{20}$  1.4504. Insol. alkalis.  $\text{HNO}_3 \rightarrow$  succinic acid and a little oxalic acid.

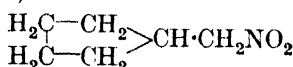
See first reference above and also Nametkin, *Chem. Zentr.*, 1912, I, 1702.

### 2-Nitro-1-methylcyclopentane.

B.p. 184-5°/758 mm. decomp., 98-9°/40 mm.  $D_4^{22}$  1.0381.  $n_D^{22}$  1.4488. Sol. alkalis.

See last reference above.

$\alpha$ -Nitromethylcyclopentane (*Cyclopentyl-nitromethane*)

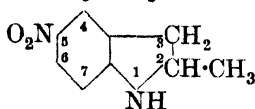


$\text{C}_6\text{H}_{11}\text{O}_2\text{N}$  MW, 129

B.p. 110°/35 mm.  $D_4^{20}$  1.0713.  $n_D^{20}$  1.4587. Sol. alkalis with yellow col.

Rosanow, *Chem. Zentr.*, 1916, I, 925.

### 5-Nitro-2-methyldihydroindole



$\text{C}_9\text{H}_{10}\text{O}_2\text{N}_2$  MW, 178

Brown needles. M.p. 82°.

N-Me:  $\text{C}_{10}\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 192. Dark red needles from EtOH. M.p. 48-9°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Less sol. ligroin.

N-Nitroso: yellow leaflets from EtOH. M.p. 135°. Very sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. hot  $\text{H}_2\text{O}$ , ligroin.

Bamberger, Sternitzki, *Ber.*, 1893, 26, 1296.

Stoermer, Dragendorff, *Ber.*, 1898, 31, 2540.

### 6-Nitro-2-methyldihydroindole.

Cryst. M.p. 50°.

B.HCl: m.p. 200° decomp.

N-Benzoyl: m.p. 137°. Spar. sol. EtOH.

N-Nitroso: m.p. 103-4°. Spar. sol. EtOH.

v. Braun, Grabowski, Rawicz, *Ber.*, 1913, 46, 3181.

### 7-Nitro-2-methyldihydroindole.

N-Nitroso: dark yellow cryst. M.p. 108°.

Stoermer, Dragendorff, *Ber.*, 1898, 31, 2540.

### 6-Nitro-3-methyldihydroindole.

Cryst. M.p. 75°.

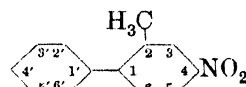
B.HCl: m.p. 192°. Mod. sol. EtOH.

N-Benzoyl: leaflets. M.p. 148°.

N-Nitroso: yellowish. M.p. 100°. Spar. sol. EtOH.

v. Braun, Grabowski, Rawicz, *Ber.*, 1913, 46, 3180.

### 4-Nitro-2-methyldiphenyl



$\text{C}_{13}\text{H}_{11}\text{O}_2\text{N}$  MW, 213

Needles from ligroin. M.p. 56-7°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Mod. sol. EtOH.

Bamberger, *Ber.*, 1895, 28, 405.

### 6-Nitro-2-methyldiphenyl.

Cryst. from EtOH. M.p. 105-6°. B.p. 155°/3 mm.

Sadler, Powell, *J. Am. Chem. Soc.*, 1934, 56, 2652.

### 2'-Nitro-2-methyldiphenyl.

Cryst. from EtOH. M.p. 64-5° (58°). B.p. 150-5°/2 mm.

See previous reference and also

Mascarelli, Gatti, *Atti accad. Lincei*, 1932, 15, 90.

### 4'-Nitro-2-methyldiphenyl.

Prisms from EtOH. M.p. 103-4°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, ligroin.

Bamberger, *Ber.*, 1895, 28, 405.

Kühling, *Ber.*, 1895, 28, 43; 1896, 29, 166.

Kliegl, Huber, *Ber.*, 1920, 53, 1647.

### 4-Nitro-3-methyldiphenyl.

Yellow liq. B.p. 195-200°/18 mm.

Grieve, Hey, *J. Chem. Soc.*, 1932, 2246.



#### 4'-Nitro-4-methyldiphenyl

##### 4'-Nitro-4-methyldiphenyl.

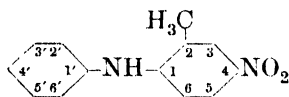
Cryst. from EtOH. M.p. 141°. Spar. sol. EtOH. Sol. hot EtOH.

Grieve, Hey, *J. Chem. Soc.*, 1932, 1891.

Gomberg, Pernert, *J. Am. Chem. Soc.*, 1926, **48**, 1379.

Carnelley, *J. Chem. Soc.*, 1876, **29**, 20.

#### 4-Nitro-2-methyldiphenylamine



$C_{13}H_{12}O_2N_2$  MW, 228

Golden-yellow plates from  $C_6H_6$ . M.p. 140–1°.

Joszt, Lésniański, *Chem. Abstracts*, 1931, **25**, 500.

#### 2'-Nitro-2-methyldiphenylamine.

Orange-yellow needles from EtOH. M.p. 76°.

McCombie, Scarborough, Waters, *J. Chem. Soc.*, 1928, 355.

#### 4'-Nitro-2-methyldiphenylamine.

Yellow leaflets from  $C_6H_6$ -ligroin. M.p. 115°. Sol. EtOH,  $C_6H_6$ . Spar. sol.  $Et_2O$ .

Ullmann, Dahmen, *Ber.*, 1908, **41**, 3749.

#### 6-Nitro-3-methyldiphenylamine.

Yellowish-red plates from EtOH, leaflets from ligroin. M.p. 110°. Sol. hot EtOH. Spar. sol. ligroin.

Schraube, Romig, *Ber.*, 1893, **26**, 581.

Borsche, *Ann.*, 1908, **359**, 76.

#### 2'-Nitro-4-methyldiphenylamine.

Orange-red needles from MeOH.Aq. M.p. 69–70°.

Jacobson, Lischke, *Ann.*, 1898, **303**, 377.

Borsche, Feise, *Ber.*, 1907, **40**, 383.

#### 4'-Nitro-4-methyldiphenylamine.

Yellow needles with blue reflex from EtOH or  $C_6H_6$ . M.p. 139°. Sol. boiling EtOH, AcOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ , ligroin.

Goldberg, D.R.P., 185,663, (*Chem. Zentr.*, 1907, II, 957).

Ullmann, Dahmen, *Ber.*, 1908, **41**, 3751.

Ullmann, D.R.P., 193,448, (*Chem. Zentr.*, 1908, I, 1003).

#### 2-Nitro-N-methyldiphenylamine.

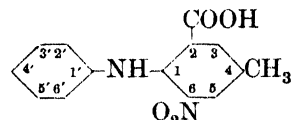
Red oil with fishy odour. B.p. 205°/15 mm.

Storrie, Tucker, *J. Chem. Soc.*, 1931, 2261.

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#### 4-Nitro-4'-methyldiphenylamine-2-carboxylic Acid

##### 6-Nitro-4-methyldiphenylamine-2-carboxylic Acid (5-Nitro-4-methyl-N-phenylanthranilic acid)



$C_{14}H_{12}O_4N_2$

MW, 272

Brick-red rhombic cryst. from EtOH. M.p. 174°.

Et ester:  $C_{16}H_{16}O_4N_2$ . MW, 300. Yellowish needles from EtOH. M.p. 136°.

Sane, Chakravarty, Parmanick, *J. Indian Chem. Soc.*, 1932, **9**, 57.

##### 4-Nitro-5-methyldiphenylamine-2-carboxylic Acid (3-Nitro-5-methyl-N-phenylanthranilic acid).

Me ester:  $C_{15}H_{14}O_4N_2$ . MW, 286. Yellow cryst. from EtOH. M.p. 84°. Sol. usual org. solvents.

Sane, Joshi, *J. Indian Chem. Soc.*, 1932, **9**, 62.

##### 4-Nitro-2'-methyldiphenylamine-2-carboxylic Acid (5-Nitro-N-o-tolylanthranilic acid).

Pale yellow needles from EtOH. M.p. 253–4°.

Locher, *Ann.*, 1894, **279**, 275.

Dey, Doraiswami, *J. Indian Chem. Soc.*, 1933, **10**, 318.

##### 5'-Nitro-2'-methyldiphenylamine-2-carboxylic Acid (N-[5-Nitro-o-tolyl]-anthranilic acid).

Cryst. from AcOH. M.p. 220–1°.

Me ester:  $C_{15}H_{14}O_4N_2$ . MW, 286. Cryst. from MeOH. M.p. 153–5°.

Weiss, Katz, *Monatsh.*, 1928, **50**, 229.

##### 4-Nitro-3'-methyldiphenylamine-2-carboxylic Acid (5-Nitro-N-m-tolylanthranilic acid).

Bright yellow needles from AcOH. M.p. 256°.

Dey, Doraiswami, *J. Indian Chem. Soc.*, 1933, **10**, 318.

##### 4-Nitro-4'-methyldiphenylamine-2-carboxylic Acid (5-Nitro-N-p-tolylanthranilic acid).

Yellow needles from AcOH. M.p. 262·5°. Sol. hot EtOH, AcOH. Spar. sol.  $Et_2O$ .

See previous reference and also

Kahn, *Ann.*, 1894, **279**, 270.

**2'-Nitro-N-methyldiphenylamine-2-carboxylic Acid**

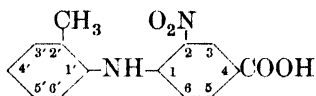
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**2' - Nitro - N - methyldiphenylamine - 2 - carboxylic Acid** (N-[2-Nitrophenyl]-N-methyl-anthranilic acid).

Deep red prisms from EtOH. M.p. 136-7° decomp.

Burton, Gibson, *J. Chem. Soc.*, 1924, 125, 2502.

**2-Nitro-2'-methyldiphenylamine-4-carboxylic Acid**



$C_{14}H_{12}O_4N_2$

MW, 272

Light brown needles. M.p. 212°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .

*Et ester*:  $C_{16}H_{16}O_4N_2$ . MW, 300. Pale yellow leaflets. M.p. 106°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .

Ullmann, *Ann.*, 1904, 332, 84.

Heidensleben, *Ber.*, 1890, 23, 3451.

**2-Nitro-4'-methyldiphenylamine-4-carboxylic Acid.**

Red needles. M.p. 257°.

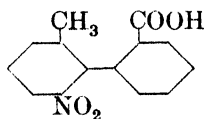
*Et ester*: dark yellow leaflets. M.p. 115°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

Heidensleben, *Ber.*, 1890, 23, 3453.

Delétra, Ullmann, *Chem. Zentr.*, 1904, I, 1569.

Schöpf, *Ber.*, 1889, 22, 3288.

**6'-Nitro-2'-methyldiphenyl-2-carboxylic Acid** (2-[6-Nitro-o-tolyl]-benzoic acid)



$C_{14}H_{11}O_4N$

MW, 257

*dl.*

Cryst. from MeOH.Aq. M.p. 173°.  $[\alpha]_D^{20} + 63.1^\circ$  in MeOH.

*Brucine salt*: cryst. +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p. 145-55°, anhyd. 172°.  $[\alpha]_D^{20} - 57.3^\circ$  in  $CHCl_3$ .

*l.*

Cryst. from MeOH.Aq. M.p. 174-5°.  $[\alpha]_D^{20} - 67.7^\circ$  in MeOH.

*Brucine salt*: cryst. +  $H_2O$  from  $H_2O$ . M.p. 169-75°, anhyd. 173-5°.  $[\alpha]_D^{20} + 30.4^\circ$  in  $CHCl_3$ .

Dict. of Org. Comp.—III.

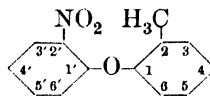
**4'-Nitro-3-methyldiphenyl Ether**

*dl.*

Pale yellow cryst. from EtOH.Aq. M.p. 171-2°. Sol. most org. solvents. Insol.  $H_2O$ .

Stoughton, Adams, *J. Am. Chem. Soc.*, 1930, 52, 5265.

**2'-Nitro-2-methyldiphenyl Ether** (o-Nitrophenyl o-tolyl ether)



$C_{13}H_{11}O_3N$

MW, 229

Red liq. B.p. 194-6°/14 mm.  $D_{20}^{20} 1.195$ . Sol. most org. solvents. Insol.  $H_2O$ .

Cook, *J. Am. Chem. Soc.*, 1901, 23, 806.

**3'-Nitro-2-methyldiphenyl Ether** (m-Nitrophenyl o-tolyl ether).

Yellow liq. B.p. 235°/14 mm.

Scarborough, Sweeten, *J. Chem. Soc.*, 1934, 53.

**4'-Nitro-2-methyldiphenyl Ether** (p-Nitrophenyl o-tolyl ether).

Yellow cryst. from pet. ether. M.p. 35°. B.p. 220-2°/27 mm. Sol. most org. solvents.

See previous reference and also

Cook, Eberly, *J. Am. Chem. Soc.*, 1902, 24, 1200.

**6-Nitro-3-methyldiphenyl Ether** (Phenyl 6-nitro-m-tolyl ether).

M.p. 63.5-64.5°.

I.G., D.R.P., 506,339, (*Chem. Abstracts*, 1931, 25, 302).

**2'-Nitro-3-methyldiphenyl Ether** (o-Nitrophenyl m-tolyl ether).

Red liq. B.p. 223°/30 mm.  $D_{27}^{27} 1.208$ . Sol. most org. solvents. Insol.  $H_2O$ .

Cook, *J. Am. Chem. Soc.*, 1901, 23, 810.

**3'-Nitro-3-methyldiphenyl Ether** (m-Nitrophenyl m-tolyl ether).

Yellow prisms from  $C_6H_6$ -pet. ether. M.p. 47°.

Scarborough, Sweeten, *J. Chem. Soc.*, 1934, 55.

**4'-Nitro-3-methyldiphenyl Ether** (p-Nitrophenyl m-tolyl ether).

Yellow cryst. from EtOH. M.p. 63° (60-1°). B.p. 230-3°/30 mm.

Cook, Frary, *Am. Chem. J.*, 1902, 28, 486.

Rarick, Brewster, Dains, *J. Am. Chem. Soc.*, 1933, 55, 1290.

**2'-Nitro-4-methyldiphenyl Ether** (*o*-Nitrophenyl *p*-tolyl ether).

Yellow cryst. from EtOH. M.p. 49°. B.p. 220°/25 mm., 210°/15 mm.

Cook, Hillyer, *Am. Chem. J.*, 1900, **24**, 526.

Cook, Sherwood, *J. Am. Chem. Soc.*, 1915, **37**, 1836.

Reilly, Drumm, Gray, *Chem. Abstracts*, 1930, **24**, 5290.

Mayer, Krieger, *Ber.*, 1922, **55**, 1661.

**3'-Nitro-4-methyldiphenyl Ether** (*m*-Nitrophenyl *p*-tolyl ether).

Pale yellow liq. B.p. 220°/23 mm.

Scarborough, Sweeten, *J. Chem. Soc.*, 1934, 56.

**4'-Nitro-4-methyldiphenyl Ether** (*p*-Nitrophenyl *p*-tolyl ether).

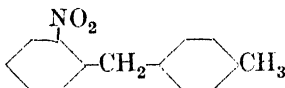
Yellow cryst. from EtOH. M.p. 69° (66°). B.p. 225°/25 mm. Sol. AcOH, C<sub>6</sub>H<sub>6</sub>.

Fries, Böker, Wallbaum, *Ann.*, 1934, **509**, 83.

Scarborough, Sweeten, *J. Chem. Soc.*, 1934, 55.

Haeussermann, Schmidt, *Ber.*, 1901, **34**, 3770.

Cook, *J. Am. Chem. Soc.*, 1903, **25**, 61.

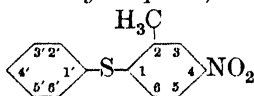
**2'-Nitro-4-methyldiphenylmethane** (2-Nitrophenyl-*p*-tolylmethane)

C<sub>14</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 227

Yellow oil. B.p. 195-8°/12 mm. Ox. → 2'-nitro-4-methylbenzophenone.

Kliegl, *Ber.*, 1908, **41**, 1847.

**4-Nitro-2-methyldiphenyl sulphide** (*Phenyl 4-nitro-o-tolyl sulphide*)

C<sub>13</sub>H<sub>11</sub>O<sub>2</sub>NS

MW, 245

Yellow plates. M.p. 82.8°. B.p. 260°/62 mm. Spar. sol. cold EtOH. Insol. H<sub>2</sub>O.

Bourgeois, Henrion, *Bull. soc. chim.*, 1932, **51**, 1421.

**2'-Nitro-2-methyldiphenyl sulphide** (*o*-Nitrophenyl *o*-tolyl sulphide).

Yellow needles from ligroin. M.p. 86-7°. Sol. EtOH, Et<sub>2</sub>O. Insol. pet. ether. Conc. H<sub>2</sub>SO<sub>4</sub> → green sol.

Mauthner, *Ber.*, 1906, **39**, 3598.

**4-Nitro-3-methyldiphenyl sulphide** (*Phenyl 4-nitro-m-tolyl sulphide*).

Pale yellow cryst. from EtOH. M.p. 72°.

Bourgeois, Henrion, *Bull. soc. chim.*, 1932, **51**, 1421.

**6-Nitro-3-methyldiphenyl sulphide** (*Phenyl 6-nitro-m-tolyl sulphide*).

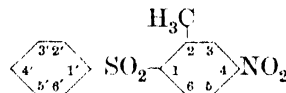
Cryst. M.p. 59.5°.

See previous reference.

**4'-Nitro-4-methyldiphenyl sulphide** (*p*-Nitrophenyl *p*-tolyl sulphide).

Yellow needles from EtOH. M.p. 81.5°.

Law, Johnson, *J. Am. Chem. Soc.*, 1930, **52**, 3625.

**4-Nitro-2-methyldiphenyl sulphone** (*Phenyl 4-nitro-o-tolyl sulphone*)

C<sub>13</sub>H<sub>11</sub>O<sub>4</sub>NS

MW, 277

Needles from EtOH. M.p. 104°.

Bourgeois, Henrion, *Bull. soc. chim.*, 1932, **51**, 1421.

**5-Nitro-2-methyldiphenyl sulphone** (*Phenyl 5-nitro-o-tolyl sulphone*).

Yellow cryst. from EtOH. M.p. 158°.

Norris, *Am. Chem. J.*, 1900, **24**, 475.

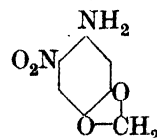
Ullmann, Lehner, *Ber.*, 1905, **38**, 736.

**2'-Nitro-4-methyldiphenyl sulphone** (*o*-Nitrophenyl *p*-tolyl sulphone).

Cryst. from AcOH. M.p. 156-7°. Easily reduced.

I.G., D.R.P., 562,824, (*Chem. Zentr.*, 1933, I, 309).

General Aniline Works, U.S.P., 1,936,721, (*Chem. Abstracts*, 1934, **28**, 1049).

**6-Nitro-3:4-methylenedioxyaniline** (5-Nitro-4-aminobenzodioxole)

C<sub>7</sub>H<sub>6</sub>O<sub>4</sub>N<sub>2</sub>

MW, 182

Needles from H<sub>2</sub>O or EtOH. M.p. 199°. Sol. warm H<sub>2</sub>O. Hot NaOH.Aq. → blood-red sol.

*N*-Me: C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub>. MW, 196. M.p. 171°.

*N*-Di-Me: C<sub>9</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 210. M.p. 98°.

*N*-Et: C<sub>9</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 210. M.p. 133°.

**6-Nitro-3 : 4-methylenedioxcinnamic Acid**

*N*-Propyl :  $C_{10}H_{12}O_4N_2$ . MW, 224. M.p. 115°.

*N*-Butyl :  $C_{11}H_{14}O_4N_2$ . MW, 238. M.p. 137°.

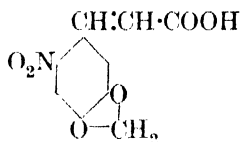
*N*-Amyl :  $C_{12}H_{16}O_4N_2$ . MW, 252. M.p. 95°.

*N*-Acetyl : bright yellow needles from AcOEt. M.p. 209°.

Jones, Robinson, *J. Chem. Soc.*, 1917, **111**, 908.

Mameli, *Gazz. chim. ital.*, 1909, **39**, ii, 182.

Parijs, *Rec. trav. chim.*, 1930, **49**, 45.

**6-Nitro-3 : 4-methylenedioxcinnamic Acid (6-Nitropiperonylideneacetic acid)**

$C_{10}H_7O_6N$

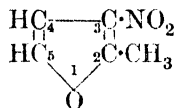
MW, 237

Yellow plates from AcOH. Decomp. at 240°. Spar. sol. EtOH,  $C_6H_6$ ,  $CHCl_3$ . Insol.  $CS_2$ , ligroin. Sol. conc.  $H_2SO_4$  with orange-red col.

*Me ester* :  $C_{11}H_9O_6N$ . MW, 251. Pale yellow needles from EtOH. M.p. 152°. Very spar. sol. cold EtOH.

*Et ester* :  $C_{12}H_{11}O_6N$ . MW, 265. Yellowish-brown needles from EtOH. M.p. 113-14°.

Feuerstein, Heimann, *J. Chem. Soc.*, 1891, **59**, 156.

**3-Nitro-2-methylfuran (3-Nitrosilvan)**

$C_5H_5O_3N$

MW, 127

Oil. M.p. 8°.

Rinkes, *Rec. trav. chim.*, 1931, **50**, 985.

**5-Nitro-2-methylfuran (5-Nitrosilvan).**

Leaflets from pet. ether. M.p. 43.5°.

Rinkes, *Rec. trav. chim.*, 1930, **49**, 1120.

Gilman, Wright, *J. Am. Chem. Soc.*, 1932, **54**, 4109.

**2-Nitro-3-methylfuran.**

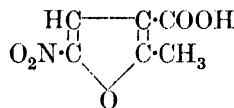
Yellow needles from pet. ether. M.p. 32.6°.

Rinkes, *Rec. trav. chim.*, 1930, **49**, 1125.

**5-Nitro-3-methylfuran.**

Leaflets from pet. ether. M.p. 29°.

Rinkes, *Rec. trav. chim.*, 1931, **50**, 988.

***p*-Nitro-β-methylhydrocinnamic Acid****5-Nitro-2-methyl-β-furoic Acid (5-Nitro-2-methylfuran-3-carboxylic acid)**

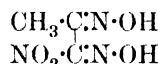
$C_8H_5O_5N$

MW, 171

Pale yellow cryst. M.p. 154-154.5°. Sublimes.

*Et ester* :  $C_8H_9O_5N$ . MW, 199. M.p. 52.5°.

Gilman, Burtner, Smith, *Rec. trav. chim.*, 1932, **51**, 407.

**Nitromethylglyoxime**

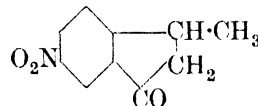
$C_3H_5O_4N_3$

MW, 147

Prisms or plates from  $Et_2O$ . M.p. 97-8° part. decomp. Very sol.  $H_2O$ , EtOH,  $Et_2O$ . Spar. sol.  $CHCl_3$ ,  $C_6H_6$ , pet. ether.

Behrend, Schmitz, *Ann.*, 1893, **277**, 320.

Behrend, Tryller, *Ann.*, 1894, **283**, 210.

**6-Nitro-3-methyl-1-hydrindone (6-Nitro-3-methylindanone-1)**

$C_{10}H_9O_3N$

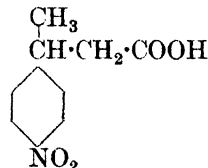
MW, 191

Cryst. from EtOH. M.p. 80°.

*Oxime* : cryst. from EtOH. M.p. 169°.

*Semicarbazone* : m.p. 253-4°. Spar. sol. EtOH.

v. Braun, Heider, *Ber.*, 1916, **49**, 1276.

***p*-Nitro-β-methylhydrocinnamic Acid (2-*p*-Nitrophenylbutyric Acid)**

$C_{10}H_{11}O_4N$

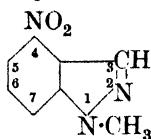
MW, 209

Cryst. from EtOH. M.p. 164°.

*Me ester* :  $C_{11}H_{13}O_4N$ . MW, 223. M.p. 63-4°.

*Chloride* :  $C_{10}H_{10}O_3NCl$ . MW, 227.5. M.p. 58°. B.p. 190-200°/17 mm.

See previous reference and also Schroeter, *Ber.*, 1907, **40**, 1596.

**4-Nitro-1-methylindazole** $C_8H_7O_2N_3$ 

MW, 177

Pale yellow needles from  $H_2O$  or pet. ether.  
M.p. 138–9°. Sol. most solvents.

Auwers, Frese, *Ber.*, 1925, 58, 1374.

**5-Nitro-1-methylindazole.**

Pale yellow needles from  $C_6H_6$ -pet. ether.  
M.p. 129°.

*Methiodide*: golden-yellow leaflets from  $H_2O$ .  
M.p. 203° decomp.

Fries, Tampke, *Ann.*, 1927, 454, 307.

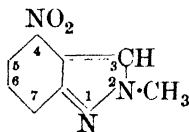
**6-Nitro-1-methylindazole.**

Yellow needles from MeOH. M.p. 108–9°.  
Sol. EtOH, hot MeOH,  $C_6H_6$ , AcOH. Mod. sol.  
hot  $H_2O$ . Spar. sol.  $Et_2O$ , pet. ether.

*Methiodide*: reddish needles from EtOH.  
M.p. 216–17°.

Auwers, Schwegler, *Ber.*, 1920, 53, 1219.

Auwers, Demuth, *Ann.*, 1927, 451, 296.

**4-Nitro-2-methylindazole** $C_8H_7O_2N_3$ 

MW, 177

Yellow needles from  $H_2O$ . M.p. 101–3°.  
Very sol. EtOH.

Auwers, Frese, *Ber.*, 1925, 58, 1374.

**5-Nitro-2-methylindazole.**

Light red needles from  $H_2O$ . M.p. 163°.

Fries, Tempke, *Ann.*, 1927, 454, 307.

**6-Nitro-2-methylindazole.**

Light yellow needles from  $C_6H_6$ . M.p. 159–  
60°.

Auwers, Schwegler, *Ber.*, 1920, 53, 1219.

Auwers, Demuth, *Ann.*, 1927, 451, 296.

**7-Nitro-2-methylindazole.**

Yellow needles. M.p. 144–5°.

Noelting, *Ber.*, 1904, 37, 2576.

**5-Nitro-4-methylindazole.**

Yellowish needles from AcOH. M.p. 259°. Spar. sol. most solvents.

Noelting, *Ber.*, 1904, 37, 2586.

**6-Nitro-4-methylindazole.**

Yellowish needles from EtOH. M.p. 177–8°.

See previous reference.

**7-Nitro-4-methylindazole.**

Golden needles from EtOH. M.p. 180–1°.

See previous reference.

**4-Nitro-5-methylindazole.**

Pale yellow needles from AcOH.Aq. M.p. 198–9°. Yellowish-red sols. in alkalis.

Noelting, *Ber.*, 1904, 37, 2590.

**6-Nitro-5-methylindazole.**

Yellow needles from  $H_2O$ . M.p. 173–4°. Sol. EtOH,  $C_6H_6$ . Very sol.  $Me_2CO$ , AcOH. Spar. sol. ligroin.

*N-Acetyl*: needles from AcOH. M.p. 182–3°.

See previous reference.

**7-Nitro-5-methylindazole.**

Pale yellow needles from EtOH. M.p. 192–5°.

Gabriel, Stelzner, *Ber.*, 1896, 29, 305.

See also previous reference.

**4-Nitro-6-methylindazole.**

Needles from  $H_2O$ . M.p. 206–7°. Sol. usual org. solvents.

Noelting, *Ber.*, 1904, 37, 2592.

**5-Nitro-6-methylindazole.**

Needles from AcOH.Aq. M.p. 231–2°.

*N-Acetyl*: needles. M.p. 203–4°. Decomp. easily.

See previous reference.

**7-Nitro-6-methylindazole.**

Yellowish needles or leaflets from  $H_2O$ , EtOH or  $C_6H_6$ . M.p. 162°. Sublimes in prisms. Volatile in steam.

See previous reference.

**4-Nitro-7-methylindazole.**

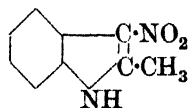
Cryst. from  $H_2O$ . M.p. 222–5°.

Noelting, *Ber.*, 1904, 37, 2587.

**6-Nitro-7-methylindazole.**

Cryst. from  $H_2O$ . M.p. 175–6°.

See previous reference.

**3-Nitro-2-methylindole** $C_9H_8O_2N_2$ 

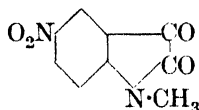
MW, 176

Yellow cryst. with violet reflex from boiling EtOH. M.p. about 248° decomp.

*N-Et*:  $C_{11}H_{12}O_2N_2$ . MW, 204. Reddish-brown needles from EtOH. M.p. 125°.

Angelico, Velardi, *Gazz. chim. ital.*, 1904, 34, ii, 61.

### 5-Nitro-1-methylisatin



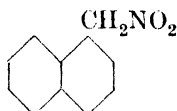
$C_9H_6O_4N_2$  MW, 206

Yellowish-red leaflets from 50% AcOH. M.p. 203°. Mod. sol. EtOH.

*Hydrazone*: yellow needles from EtOH. M.p. 210° decomp.

Borsche, Hildegard, Weissmann, Fritzsche, *Ber.*, 1924, 57, 1151.

**1- $\omega$ -Nitromethylnaphthalene** ( $\alpha$ -Naphthyl-nitromethane)

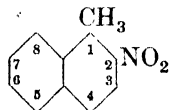


$C_{11}H_9O_2N$  MW, 187

Yellowish needles from pet. ether. M.p. 72-3°.

Wislicenus, Wren, *Ber.*, 1905, 38, 508.

### 2-Nitro-1-methylnaphthalene



$C_{11}H_9O_2N$  MW, 187

Yellow needles from EtOH. M.p. 58-9°.

Vesely, Štursa, Olejníček, Rein, *Chem. Zentr.*, 1930, I, 2734.

### 4-Nitro-1-methylnaphthalene.

Pale yellow needles from EtOH. M.p. 71-2°. B.p. 182-3°/18 mm., 176°/12 mm. Sol. usual org. solvents. Conc.  $H_2SO_4 \rightarrow$  red sol. Dil.  $HNO_3 \rightarrow$  4-nitro-1-naphthoic acid.

Lesser, *Ann.*, 1913, 402, 11.

See also previous reference.

### 5-Nitro-1-methylnaphthalene.

Brownish needles from EtOH. M.p. 82-3°.

Vesely, Štursa, Olejníček, Rein, *Chem. Zentr.*, 1930, I, 2734.

### 6-Nitro-1-methylnaphthalene.

Yellow needles from EtOH. M.p. 76-7°.

See previous reference.

### 7-Nitro-1-methylnaphthalene.

Yellow needles from EtOH. M.p. 98-9°.

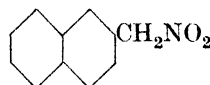
See previous reference.

### 8-Nitro-1-methylnaphthalene.

Brownish leaflets from EtOH. M.p. 63-4°.

See previous reference.

**2- $\omega$ -Nitromethylnaphthalene** ( $\beta$ -Naphthyl-nitromethane)

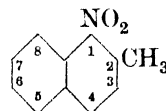


$C_{11}H_9O_2N$  MW, 187

Cryst. M.p. 72° (not sharp). Very sol. org. solvents. Decomp. above m.p.

Wislicenus, Wren, *Ber.*, 1905, 38, 510.

### 1-Nitro-2-methylnaphthalene



$C_{11}H_9O_2N$  MW, 187

Yellow needles from EtOH. M.p. 81°. B.p. 185-6°/18 mm.

Lesser, *Ann.*, 1913, 402, 4, 31.

Vesely, Rein, *Chem. Zentr.*, 1929, II, 1669.

### 3-Nitro-2-methylnaphthalene.

Yellow plates from EtOH. M.p. 117-18°.

Vesely, Štursa, *Chem. Zentr.*, 1934, I, 3589.

### 5-Nitro-2-methylnaphthalene.

Yellow needles from EtOH. M.p. 61-2°.

Vesely, Páč, *Chem. Zentr.*, 1930, II, 1548.

### 6-Nitro-2-methylnaphthalene.

Yellow needles from EtOH. M.p. 119°.

See previous reference.

### 7-Nitro-2-methylnaphthalene.

Yellow cryst. from EtOH. M.p. 102°.

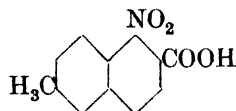
See previous reference.

### 8-Nitro-2-methylnaphthalene.

Yellow needles from EtOH. M.p. 36-8°.

See previous reference.

### 1-Nitro-6-methyl-2-naphthoic Acid



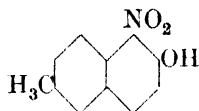
$C_{12}H_9O_4N$

MW, 231

Needles from AcOH. M.p. 238-9°.

Mayer, Alken, *Ber.*, 1932, 55, 2282.

### 1-Nitro-6-methyl-2-naphthol



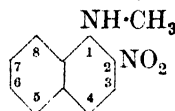
$C_{11}H_9O_3N$

MW, 203

Yellow needles from EtOH.Aq. M.p. 81-2°. Sol. most. org. solvents. Dil. NaOH  $\longrightarrow$  blood-red sol.

Dziewoński, Schoenówna, Waldmann, *Ber.*, 1925, 58, 1215.

### 2-Nitro-N-methyl-1-naphthylamine



$C_{11}H_{10}O_2N_2$

MW, 202

Dark red needles from pet. ether. M.p. 114°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Insol. H<sub>2</sub>O.

Hoogeveen, *Rec. trav. chim.*, 1931, 50, 37.

### 4-Nitro-N-methyl-1-naphthylamine.

Orange plates. M.p. 184-5°.

Veselý, Vojtěch, *Chem. Zentr.*, 1929, II, 425.

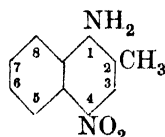
### 8-Nitro-N-methyl-1-naphthylamine.

Cryst. M.p. 81°.

N-Benzenesulphonyl: yellow needles from EtOH. M.p. 179°.

See previous reference.

### 4-Nitro-2-methyl-1-naphthylamine



$C_{11}H_{10}O_2N_2$

MW, 202

Orange needles from C<sub>6</sub>H<sub>6</sub>. M.p. 184-5°. Very sol. EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>.

N-Acetyl: pale yellow needles from EtOH. M.p. 240-1°.

Veselý, Kapp, *Chem. Zentr.*, 1924, II, 2751.

### 2-Nitro-4-methyl-1-naphthylamine.

Cryst. M.p. 179-5°.

N-Acetyl: m.p. 224-5°.

Veselý, Štursa, Olejníček, Rein, *Chem. Abstracts*, 1930, 24, 611.

### 3-Nitro-4-methyl-1-naphthylamine.

Red plates. M.p. 131-2°.

N-Acetyl: yellowish needles. M.p. 230-1°.

Veselý, Štursa, Olejníček, Rein, *Chem. Abstracts*, 1930, 24, 3008.

### 2-Nitro-5-methyl-1-naphthylamine.

Cryst. M.p. 178-9°. Insol. 62% H<sub>2</sub>SO<sub>4</sub>.

N-Acetyl: needles from AcOEt. M.p. 245-6°.

Veselý, Štursa, Olejníček, Rein, *Chem. Abstracts*, 1930, 24, 611.

### 4-Nitro-5-methyl-1-naphthylamine.

Cryst. M.p. 163-4°. Sol. 62% H<sub>2</sub>SO<sub>4</sub>.

N-Acetyl: prisms from AcOEt. M.p. 197-8°.

See previous reference.

### 2-Nitro-6-methyl-1-naphthylamine.

Brownish-red needles from EtOH. M.p. 171°.

N-Acetyl: yellow needles from EtOH. M.p. 210-11°.

Veselý, Páč, *Chem. Zentr.*, 1930, II, 1548.

### 4-Nitro-6-methyl-1-naphthylamine.

Yellow or orange needles. M.p. 167-9°.

N-Acetyl: golden-yellow needles from EtOH. M.p. 202°.

See previous reference.

### 5-Nitro-6-methyl-1-naphthylamine.

Red plates from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 134-6°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>.

N-Acetyl: yellow needles from EtOH. M.p. 192°.

B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>: m.p. 270° decomp.

Veselý, Kapp, *Chem. Zentr.*, 1924, II, 2751.

Giral, *Chem. Zentr.*, 1934, II, 940.

### 2-Nitro-7-methyl-1-naphthylamine.

Red needles from EtOH. M.p. 185°.

N-Acetyl: yellow needles from AcOEt. M.p. 219-20°.

Veselý, Páč, *Chem. Zentr.*, 1930, II, 1548.

### 4-Nitro-7-methyl-1-naphthylamine.

Orange needles. M.p. 183°.

N-Acetyl: yellow needles from AcOH-AcOEt. M.p. 229-30°.

See previous reference.

### 8-Nitro-7-methyl-1-naphthylamine.

Cryst. from EtOH. M.p. 106-7°.

N-Acetyl: m.p. 191-3°.

B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>: m.p. 175-80°.

Veselý, Rein, *Chem. Abstracts*, 1928, 22, 1352.

Giral, *Chem. Zentr.*, 1934, II, 940.

**2-Nitro-8-methyl-1-naphthylamine.**

Brownish-red needles from EtOH. M.p. 150–2°.

N-Acetyl: cryst. from EtOH. M.p. 186–7°.

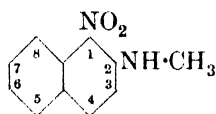
Veselý, Štursa, Olejníček, Rein, *Chem. Zentr.*, 1930, I, 2735.

**4-Nitro-8-methyl-1-naphthylamine.**

Orange needles. M.p. 162–3°.

N-Acetyl: yellow cryst. from AcOH. M.p. 193–4°.

See previous reference.

**1-Nitro-N-methyl-2-naphthylamine**

$C_{11}H_{10}O_2N_2$

MW, 202

Red needles from AcOH. M.p. 124–5°.

N-Acetyl: greenish-yellow cryst. from EtOH. M.p. 112–13°.

N-Benzenesulphonyl: needles from EtOH. M.p. 158–9°.

N-Nitroso: needles from EtOH. M.p. 100°.

Meldola, Lane, *J. Chem. Soc.*, 1904, 85, 1602.

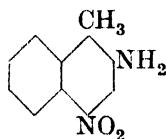
**6-Nitro-N-methyl-2-naphthylamine.**

Golden-yellow plates from EtOH. M.p. 185–6°.

N-Acetyl: needles. M.p. 186–7°.

Picrate: m.p. 138–40°.

Veselý, Vojtěch, *Chem. Zentr.*, 1929, II, 425.

**4-Nitro-1-methyl-2-naphthylamine**

$C_{11}H_{10}O_2N_2$

MW, 202

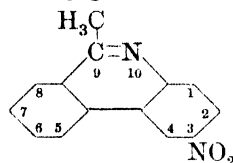
Orange needles from EtOH. M.p. 126–8°.

N-Acetyl: yellow needles from EtOH. M.p. 203–4°.

Veselý, Štursa, Olejníček, Rein, *Chem. Abstracts*, 1930, 24, 3008.

**4-Nitro-3-methyl-1-p-nitrophenylpyrazolone-5.**

See Picrolonic Acid.

**3-Nitro-9-methylphenanthridine**

$C_{14}H_{10}O_2N_2$

MW, 238

Brown prisms from  $C_6H_6$ . M.p. 201°. Sol. dil. min. acids. Almost insol. dil. AcOH. More sol. in  $C_6H_6$  than in EtOH.

Morgan, Walls, *J. Chem. Soc.*, 1932, 2228.

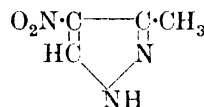
**7-Nitro-9-methylphenanthridine.**

Buff needles from  $C_6H_6$ . M.p. 243–5°. More sol. in  $C_6H_6$  than in EtOH.

See previous reference.

**Nitromethylphenylcinnamic Acid.**

See Nitromethylstilbene- $\alpha$ -carboxylic Acid.

**4-Nitro-3-methylpyrazole (4-Nitro-5-methylpyrazole)**

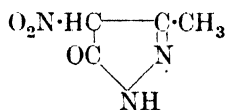
$C_4H_5O_2N_3$

MW, 127

Prisms from  $H_2O$ . M.p. 134°. B.p. 325°/748 mm.

Knorr, *Ann.*, 1894, 279, 228.

Viguier, *Ann. chim. phys.*, 1913, 28, 469.

**4-Nitro-3-methylpyrazolone-5 (4-Nitro-5-hydroxy-3-methylpyrazole)**

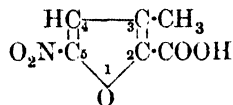
$C_4H_5O_3N_3$

MW, 143

Cryst. from AcOH. M.p. 276°. Spar. sol. EtOH,  $Me_2CO$ , amyl alcohol. Insol. pet. ether. Reacts acid.

Betti, Niccoli, *Gazz. chim. ital.*, 1904, 34, 186.

Bülow, Haas, *Ber.*, 1910, 43, 2655.

**5-Nitro-3-methylpyromucic Acid (5-Nitro-3-methylfuran-2-carboxylic acid, 5-nitro-3-methyl- $\alpha$ -furoic acid, 5-nitroelsholtzic acid)**

$C_6H_5O_5N$

MW, 171



#### 4-Nitro-5-methylpyromucic Acid

Cryst. from  $C_6H_6$ . M.p.  $160^\circ$ .  
*Ester*:  $C_8H_9O_5N$ . MW, 199. Yellow cryst.  
from  $C_6H_6$ -pet. ether. M.p.  $61^\circ$ .

Rinkes, *Rec. trav. chim.*, 1931, **50**, 981.

**4-Nitro-5-methylpyromucic Acid** (4-Nitro-5-methylfuran-2-carboxylic acid, 4-nitro-5-methyl- $\alpha$ -furoic acid).

Needles from Py. M.p.  $159-60^\circ$ .

*Me ester*:  $C_7H_7O_5N$ . MW, 185. M.p.  $81^\circ$ .

Rinkes, *Rec. trav. chim.*, 1930, **49**, 1118.

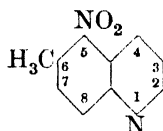
**Nitro-2-methylquinoline.**

See Nitroquinaldine.

**Nitro-4-methylquinoline.**

See Nitrolepidine.

**5-Nitro-6-methylquinoline**



$C_{10}H_8O_2N_2$

MW, 188

Pale yellow needles from EtOH. M.p.  $116-17^\circ$ . Sol. usual solvents. Insol. cold  $H_2O$ . Weak base.  $Fe + AcOH \rightarrow$  5-amino-6-methylquinoline.

*Methiodide*: cryst. M.p.  $189-90^\circ$ . Sol.  $H_2O$ . Spar. sol. EtOH. Bitter taste.

Noelting, Trautmann, *Ber.*, 1890, **23**, 3655.

Bogert, Fisher, *J. Am. Chem. Soc.*, 1912, **34**, 1570.

**8-Nitro-6-methylquinoline.**

Pale yellow needles from  $H_2O$ . M.p.  $122^\circ$ . Sol. usual solvents.

Bartow, McCollum, *J. Am. Chem. Soc.*, 1904, **26**, 702.

See also previous references.

**5-Nitro-8-methylquinoline.**

Pale yellow needles from EtOH. M.p.  $93^\circ$ . Sol. usual solvents.  $Fe + AcOH \rightarrow$  5-amino-8-methylquinoline. Warm KOH in dil. EtOH  $\rightarrow$  yellow col. changing through green to red.

Noelting, Trautmann, *Ber.*, 1890, **23**, 3673.

**6-Nitro-8-methylquinoline.**

Cryst. from EtOH. M.p.  $129^\circ$ . Sol. EtOH. Spar. sol.  $H_2O$ .

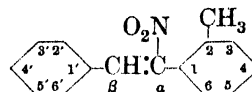
Lellmann, Ziemssen, *Ber.*, 1891, **24**, 2116.

**Nitro-N-methyl- $\alpha$ -quinolone.**

See under Nitrocarbostyrl.

#### 200 6-Nitro-3-methylstilbene- $\alpha$ -carboxylic Acid

**$\alpha$ -Nitro-2-methylstilbene**



$C_{15}H_{13}O_2N$

MW, 239

Yellow plates from EtOH. M.p.  $92^\circ$ . Sol.  $C_6H_6$ ,  $CHCl_3$ ,  $CS_2$  in cold, MeOH, EtOH,  $Et_2O$ , AcOH in hot.

Meisenheimer, Beisswenger, Kauffmann, Kummer, Link, *Ann.*, 1929, **468**, 202.

**$\beta$ -Nitro-2-methylstilbene.**

Needles from EtOH. M.p.  $99^\circ$ .

See previous reference.

**$\alpha$ -Nitro-3-methylstilbene.**

Yellow plates from MeOH or EtOH. M.p.  $82^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , Py.

See previous reference.

**$\beta$ -Nitro-3-methylstilbene.**

Yellow needles from MeOH. M.p.  $51^\circ$ . B.p.  $195^\circ/14$  mm. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. AcOH.

See previous reference.

**2'-Nitro-4-methylstilbene.**

Red prisms from EtOH. M.p.  $211^\circ$ .

Pschorr, *Ber.*, 1906, **39**, 3112.

**4'-Nitro-4-methylstilbene.**

Greenish-yellow leaflets from EtOH. M.p.  $150^\circ$ . Sol.  $Et_2O$ , hot EtOH,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Less sol. ligroin.

Pfeiffer, *Ber.*, 1915, **48**, 1792.

**$\alpha$ -Nitro-4-methylstilbene.**

Yellow prisms from EtOH. M.p.  $75-6^\circ$ .

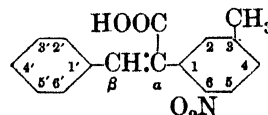
Meisenheimer, Beisswenger, Kauffmann, Kummer, Link, *Ann.*, 1929, **468**, 202.

**$\beta$ -Nitro-4-methylstilbene.**

Yellow needles or leaflets from MeOH. M.p.  $79^\circ$ .

See previous reference.

**6-Nitro-3-methylstilbene- $\alpha$ -carboxylic Acid** ( $\alpha$ -[6-Nitro-3-methylphenyl]-cinnamic acid)



$C_{16}H_{13}O_4N$

MW, 283

Yellowish cryst. from  $C_6H_6$ -ligroin. M.p.  $202-3^\circ$ . Spar. sol.  $C_6H_6$ , AcOH. Insol. ligroin.

Mayer, Balle, *Ann.*, 1914, **403**, 193.

**2'-Nitro-3-methylstilbene- $\alpha$ -carboxylic Acid** 201

**2'-Nitro-3-methylstilbene- $\alpha$ -carboxylic Acid** (2-Nitro- $\alpha$ -[3-methylphenyl]-cinnamic acid). Cryst. from  $C_6H_6$ -ligroin. M.p. 141-2°. *Me ester*:  $C_{17}H_{15}O_4N$ . MW, 297. Yellowish needles from ligroin. M.p. 70-1°.

See previous reference.

**2-Nitro-3'-methylstilbene- $\alpha$ -carboxylic Acid.**

Prisms from AcOH. M.p. 180-1°. Spar. sol.  $C_6H_6$ , AcOH.

See previous reference.

**2'-Nitro-3'-methylstilbene- $\alpha$ -carboxylic Acid** (2-Nitro-3-methyl- $\alpha$ -phenylcinnamic acid).

Prisms from  $C_6H_6$ -ligroin. M.p. 221°. Spar. sol. MeOH,  $C_6H_6$ . Insol. ligroin.

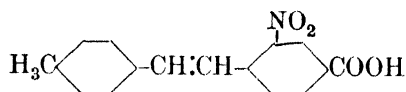
See previous reference.

**6'-Nitro-3'-methylstilbene- $\alpha$ -carboxylic Acid** (6-Nitro-3-methyl- $\alpha$ -phenylcinnamic acid).

Prisms from  $C_6H_6$ -ligroin. M.p. 203-4°. Spar. sol. AcOH,  $C_6H_6$ . Insol. ligroin.

See previous reference.

**2-Nitro-4'-methylstilbene-4-carboxylic Acid**



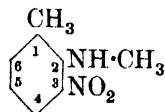
$C_{16}H_{13}O_4N$  MW, 283

*Et ester*:  $C_{18}H_{17}O_4N$ . MW, 311. Yellow needles from EtOH. M.p. 99-100°.

*Nitrile*:  $C_{16}H_{12}O_2N_2$ . MW, 264. Yellow needles from AcOH. M.p. 170°.

Pfeiffer, *Ann.*, 1916, 411, 141.

**3-Nitro-N-methyl-o-toluidine**



$C_8H_{10}O_2N_2$  MW, 166

Cryst. M.p. 48°.

Gnehm, Blumer, *Ann.*, 1899, 304, 98.

**4-Nitro-N-methyl-o-toluidine.**

Red leaflets from EtOH, yellow needles from ligroin. M.p. 107.5°. Sol.  $CHCl_3$ ,  $Et_2O$ . Spar. sol. EtOH, ligroin.

*N-Acetyl*: yellow leaflets from EtOH.Aq. M.p. 119°.

Vorländer, Siebert, *Ber.*, 1919, 52, 300.

See also previous reference.

**N'-Nitro-N-methylurea**

**5-Nitro-N-methyl-o-toluidine.**

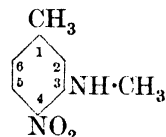
Yellow leaflets from EtOH. M.p. 137°.

*N-Acetyl*: rhombic cryst. from EtOH.Aq. M.p. 97°. Very sol. EtOH.

Bernthsen, *Ber.*, 1892, 25, 3131.

Kock, *Ann.*, 1888, 243, 309.

**4-Nitro-N-methyl-m-toluidine**



$C_8H_{10}O_2N_2$  MW, 166

Brownish-yellow prisms from MeOH.Aq. M.p. 83°.

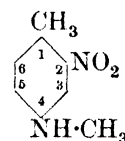
Fischer, Rigaud, *Ber.*, 1902, 35, 1259.

**6-Nitro-N-methyl-m-toluidine.**

Yellowish-brown plates with blue reflex. M.p. 92-3°.

Stoermer, Hoffmann, *Ber.*, 1898, 31, 2535.

**2-Nitro-N-methyl-p-toluidine**



$C_8H_{10}O_2N_2$  MW, 166

Red needles or prisms from EtOH. M.p. 57°.

*N-Acetyl*: light yellow needles from EtOH. M.p. 128-128.5°.

Pinnow, *Ber.*, 1895, 28, 3040.

**3-Nitro-N-methyl-p-toluidine.**

Red needles from EtOH. M.p. 84-5°.

*N-Acetyl*: plates. M.p. 64°. B.p. 250-5°/270 mm. Very sol. usual solvents. *Picrate*: yellow cryst. M.p. 210-12°.

Morgan, Jobling, Barnett, *J. Chem. Soc.*, 1912, 101, 1212.

Gattermann, *Ber.*, 1885, 18, 1487.

Niementowski, *Ber.*, 1887, 20, 1876.

**N'-Nitro-N-methylurea**

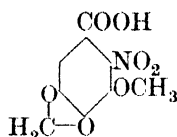


$C_2H_5O_3N_3$  MW, 119

Needles from  $C_6H_6$ . M.p. 159° decomp. Very sol.  $Me_2CO$ ,  $AcOEt$ . Sol. EtOH. Mod. sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ ,  $Et_2O$ .

Backer, *Rec. trav. chim.*, 1915, 34, 188.

## 2-Nitromyristic Acid

 $C_9H_7O_7N$ 

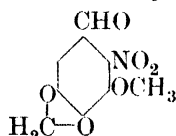
MW, 241

Needles from  $H_2O$ . M.p.  $245^\circ$ . Sol. EtOH, AcOH, AcOEt. Spar. sol. hot  $H_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Turns yellow on exposure to light.

*Et ester*:  $C_{11}H_{11}O_7N$ . MW, 269. Prisms from EtOH. M.p.  $82^\circ$ . Turns yellow on exposure to light.

Salway, *J. Chem. Soc.*, 1909, **95**, 1165; 1911, **99**, 267.

## 2-Nitromyristinaldehyde

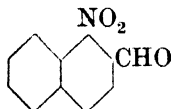
 $C_9H_7O_6N$ 

MW, 225

Needles from EtOH. M.p.  $131-2^\circ$ . Sol. most org. solvents. Spar. sol. pet. ether. Turns deep yellow on exposure to light.  $KMnO_4 \rightarrow$  nitromyristic acid.

Salway, *J. Chem. Soc.*, 1909, **95**, 1160.

## 1-Nitro-2-naphthaldehyde

 $C_{11}H_7O_3N$ 

MW, 201

Leaflets from ligroin. M.p.  $99^\circ$ . Volatile in steam. Forms bisulphite comp.

Mayer, Oppenheimer, *Ber.*, 1918, **51**, 1241.

## 1-Nitronaphthalene

 $C_{10}H_7O_2N$ 

MW, 173

Yellow needles from EtOH. M.p.  $61.5^\circ$  ( $58.5^\circ$ ). B.p.  $304^\circ$ .  $D_4$  1.331. Very sol.  $CS_2$ . Conc.  $H_2SO_4 \rightarrow$  dark red sol.

*Picrate*: m.p.  $71^\circ$ .

Beilstein, Kuhlberg, *Ann.*, 1873, **169**, 89.

Fichter, Plüss, Swiss P., 150,298, (*Chem.*

*Abstracts*, 1932, **26**, 4830).

## 2-Nitronaphthalene.

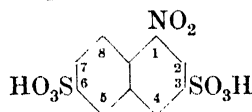
Plates from EtOH.Aq. M.p.  $79^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH. Volatile in steam.  $Zn + AcOH \rightarrow$  2-naphthylamine.

Meisenheimer, Witte, *Ber.*, 1903, **36**, 4157.

du Pont, U.S.P., 1,836,211, (*Chem. Abstracts*, 1932, **26**, 995).

Nitronaphthalene-1:8-dicarboxylic Acid.  
See Nitronaphthalic Acid.

## 1 - Nitronaphthalene - 3 : 6 - disulphonic Acid (4-Nitronaphthalene-2 : 7-disulphonic acid)

 $C_{10}H_7O_8NS_2$ 

MW, 333

Needles. Very sol.  $H_2O$ . Sol. EtOH. Insol.  $Et_2O$ .

*Dichloride*:  $C_{10}H_5O_6NCl_2S_2$ . MW, 370. Yellow needles from AcOH. M.p.  $140-1^\circ$ . Sol.  $CHCl_3$ , AcOH,  $C_6H_6$ , ligroin. Mod. sol.  $Et_2O$ ,  $CS_2$ . Dist. with  $PCl_5 \rightarrow$  1 : 3 : 6-trichloronaphthalene.

*Diamide*:  $C_{10}H_9O_6N_3S_2$ . MW, 331. Needles. M.p.  $286-7^\circ$  decomp. Sol. EtOH. Very spar. sol.  $H_2O$ . Sol.  $NH_3$ .Aq.

Alén, *Ber.*, 1884, **17**, 435 (*Ref.*).

Armstrong, Wynne, *Chem. News*, 1895, **71**, 254.

## 1 - Nitronaphthalene - 3 : 7 - disulphonic Acid (4-Nitronaphthalene-2 : 6-disulphonic acid).

Cryst. Very sol.  $H_2O$ . Sol. EtOH.

*Dichloride*: prisms from  $C_6H_6$ . M.p.  $190-2^\circ$ . Mod. sol. AcOH,  $C_6H_6$ . Spar. sol.  $Et_2O$ ,  $CS_2$ .  $PCl_5$  at  $200^\circ \rightarrow$  1 : 3 : 7-trichloronaphthalene.

*Diamide*: needles from  $H_2O$ . Does not melt below  $300^\circ$ . Spar. sol.  $H_2O$ . Very spar. sol. EtOH.

Alén, *Ber.*, 1884, **17**, 437 (*Ref.*).

Armstrong, Wynne, *Chem. News*, 1890, **61**, 93.

## Nitronaphthalene-disulphonic Acid.

The following Nitronaphthalene disulphonic Acids, for which there is little or no data, have also been prepared :

## 1-Nitronaphthalene-3 : 8-disulphonic Acid (8-Nitronaphthalene-1 : 6-disulphonic acid).

Friedländer, *Ber.*, 1895, **28**, 1535.

Armstrong, Wynne, *Chem. News*, 1891, **63**, 124.

Ewer, Pick, D.R.P., 52,724.

1-Nitronaphthalene-4 : 8-disulphonic Acid (4-Nitronaphthalene-1 : 5-disulphonic acid).

Friedländer, Karamessinis, Schenk, *Ber.*, 1922, **55**, 48.

Cassella, D.R.P., 65,997.

1-Nitronaphthalene-5 : 8-disulphonic Acid (5-Nitronaphthalene-1 : 4-disulphonic acid).

Gattermann, *Ber.*, 1899, **32**, 1156.

Bayer, D.R.P., 70,857.

2-Nitronaphthalene-4 : 8-disulphonic Acid (3-Nitronaphthalene-1 : 5-disulphonic acid).

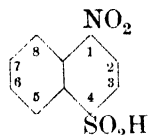
Friedländer, Karamessinis, Schenk, *Ber.*, 1922, **55**, 47.

Tinker, Hansen, U.S.P., 1,836,204, (*Chem. Abstracts*, 1932, **26**, 998).

Cotton, U.S.P., 1,756,537, (*Chem. Abstracts*, 1930, **24**, 3023).

I.G., F.P., 734,616, (*Chem. Abstracts*, 1933, **27**, 1002).

#### 1-Nitronaphthalene-4-sulphinic Acid



$C_{10}H_7O_4NS$

MW, 237

Yellow needles from  $H_2O$ . M.p.  $131^\circ$ . Sol. EtOH, Et<sub>2</sub>O, AcOH, AcOEt, Me<sub>2</sub>CO. Spar. sol.  $CHCl_3$ ,  $C_6H_6$ .

Brunetti, *J. prakt. Chem.*, 1930, **128**, 44.

#### 1-Nitronaphthalene-5-sulphinic Acid.

Cryst. M.p.  $140^\circ$ .

*Na salt* : yellow leaflets from  $H_2O$ .

Reissert, *Ber.*, 1922, **55**, 863.

#### 1-Nitronaphthalene-6-sulphinic Acid.

Prisms with no m.p.

Vorozhtzov, Gribov, *Chem. Abstracts*, 1933, **27**, 2440.

#### 1-Nitronaphthalene-7-sulphinic Acid.

Yellow needles from  $H_2O$ . Decomp. at  $150-5^\circ$  without melting.

See previous reference.

#### 1-Nitronaphthalene-8-sulphinic Acid.

Cryst. Deflagrates at  $110^\circ$ . Turns brown in air. Heat with 60%  $H_2SO_4$  in steam  $\rightarrow$  1-nitronaphthalene.

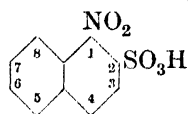
*K salt* : golden leaflets +  $2H_2O$ . Very sol.  $H_2O$ .

*Ba salt* : leaflets +  $6H_2O$ . Very sol.  $H_2O$ .

Erdmann, Süvern, *Ann.*, 1893, **275**, 306.

Reissert, *Ber.*, 1922, **55**, 862.

#### 1-Nitronaphthalene-2-sulphonic Acid



$C_{10}H_7O_5NS$

MW, 253

Green cryst. +  $H_2O$ . M.p.  $104.7^\circ$ . Deliquescent.

*Na salt* : cryst. +  $4\frac{1}{2}H_2O$ . M.p.  $93-4^\circ$ .

*Chloride* :  $C_{10}H_6O_4NClS$ . MW, 271.5. Pink needles from  $C_6H_6$ -pet. ether. M.p.  $120.5^\circ$ .

*Amide* :  $C_{10}H_8O_4N_2S$ . MW, 252. Needles from 50% EtOH. M.p.  $214.3^\circ$ .

Vorozhtzov, Kozlov, *Chem. Abstracts*, 1933, **27**, 2441.

#### 1-Nitronaphthalene-3-sulphonic Acid.

*K salt* : needles. Spar. sol.  $H_2O$ .

*Et ester* :  $C_{12}H_{11}O_5NS$ . MW, 281. Yellow needles from EtOH. M.p.  $114.5^\circ$ . Spar. sol. cold EtOH.

*Chloride* : yellow needles. M.p.  $139.5-140^\circ$ . Very spar. sol. AcOH. Excess  $PCl_5 \rightarrow$  1 : 3-dichloronaphthalene.

*Amide* : needles. M.p.  $225^\circ$ .

Cleve, *Ber.*, 1886, **19**, 2179.

Armstrong, Wynne, *Chem. News*, 1889, **59**, 95.

Erdmann, Süvern, *Ann.*, 1893, **275**, 252.

#### 1-Nitronaphthalene-4-sulphonic Acid.

Cryst. Readily sol.  $H_2O$ .

*Na salt* : needles +  $H_2O$ . Very sol.  $H_2O$ .

*K salt* : needles. Spar. sol. cold  $H_2O$ .

*Ca salt* : leaflets +  $2H_2O$ . Sol. 37 parts  $H_2O$  at  $17^\circ$  and 16 parts boiling.

*Ba salt* : needles +  $H_2O$ . Sol. 66 parts cold and 33 parts boiling  $H_2O$ .

*Me ester* :  $C_{11}H_9O_5NS$ . MW, 267. Needles. M.p.  $117^\circ$ . Spar. sol. EtOH.

*Et ester* : prisms from EtOH. M.p.  $93^\circ$ .

*Chloride* : prisms from  $CHCl_3$ . M.p.  $99^\circ$ .

*Amide* : cryst. M.p.  $188^\circ$ .

Cleve, *Ber.*, 1890, **23**, 958.

Brunetti, *J. prakt. Chem.*, 1930, **128**, 46.

#### 1-Nitronaphthalene-5-sulphonic Acid.

Prisms +  $4H_2O$  from  $H_2O$ . Very sol.  $H_2O$ . Sol. EtOH. Spar. sol Et<sub>2</sub>O, dil.  $H_2SO_4$ . Very bitter taste.  $NaHg \rightarrow$  1-naphthylamine.

*NH<sub>4</sub> salt* : needles +  $1\frac{1}{2}H_2O$ . Readily sol.  $H_2O$ .

*Na salt* : plates +  $\frac{1}{2}H_2O$ . Very sol.  $H_2O$ .

*K salt* : plates +  $1H_2O$  from  $H_2O$ . Sol. 25 parts  $H_2O$  at  $17^\circ$ .

*Ca salt* : leaflets +  $2H_2O$  from  $H_2O$ . 100 c.c.  $H_2O$  dissolve 0.34 g. at  $16^\circ$  and 7.5 g. at  $100^\circ$ .

*Ba salt*: needles +  $3\text{H}_2\text{O}$ . Spar sol. cold  $\text{H}_2\text{O}$ .

*Me ester*: prisms from  $\text{CHCl}_3$ . M.p.  $117.5^\circ$ .

*Et ester*: needles from  $\text{CHCl}_3$ -pet. ether. M.p.  $101-2^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Spar sol.  $\text{Et}_2\text{O}$ , cold  $\text{EtOH}$ .

*Chloride*: needles from  $\text{AcOH}$  or  $\text{Et}_2\text{O}$ . M.p.  $113^\circ$ . Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ . Insol. pet. ether.

*Amide*: yellowish prisms from  $\text{EtOH}$ . M.p.  $236^\circ$  decomp. ( $225^\circ$ ). Spar. sol.  $\text{Et}_2\text{O}$ , cold  $\text{EtOH}$ . Insol.  $\text{H}_2\text{O}$ .

*Anilide*: cryst. from 90%  $\text{EtOH}$ . M.p.  $123^\circ$ .

Erdmann, *Ann.*, 1888, **47**, 311.

Cleve, *Ber.*, 1890, **23**, 958.

Erdmann, Süvern, *Ann.*, 1893, **275**, 246.

Steiger, *Helv. Chim. Acta*, 1933, **16**, 798.

Vorozhtzov, Gribov, *Chem. Abstracts*, 1933, **27**, 2440.

### 1-Nitronaphthalene-6-sulphonic Acid.

Yellow needles +  $2\text{H}_2\text{O}$  from conc.  $\text{HCl}$ . M.p.  $118-19^\circ$ . Bitter taste. Very sol.  $\text{H}_2\text{O}$ . Sol.  $\text{EtOH}$ .

*NH<sub>4</sub> salt*: yellow plates. Sol. 13 parts cold  $\text{H}_2\text{O}$ .

*Na salt*: yellow cryst. +  $3\text{H}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$ .

*K salt*: plates. Sol. to 3.5% in  $\text{H}_2\text{O}$  at  $20^\circ$ .

*Cu salt*:  $\text{Cu}(\text{C}_{10}\text{H}_6\text{O}_5\text{NS})_2 \cdot 6\text{H}_2\text{O}$ . Green needles. Mod. sol. hot  $\text{H}_2\text{O}$ . Loses  $4\text{H}_2\text{O}$  at  $100^\circ$ .

*Ba salt*: yellow leaflets +  $\text{H}_2\text{O}$ . Sol. 782 parts  $\text{H}_2\text{O}$  at  $22^\circ$ .

*Et ester*: yellowish needles from  $\text{C}_6\text{H}_6$ . M.p.  $114^\circ$ .

*Chloride*: prisms from  $\text{C}_6\text{H}_6$ . M.p.  $125.5^\circ$ . Excess  $\text{PCl}_5 \rightarrow$  1:6-dichloronaphthalene.

*Amide*: yellow cryst. powder. M.p.  $184^\circ$  ( $180^\circ$ ). Mod. sol. boiling  $\text{EtOH}$ . Very spar. sol. boiling  $\text{H}_2\text{O}$ .

Armstrong, Wynne, *Ber.*, 1891, **24**, 654.

Palmaer, *Ber.*, 1888, **21**, 3260.

Cleve, *Ber.*, 1886, **19**, 2179.

Armstrong, *Chem. News*, 1889, **59**, 95.

Erdmann, Süvern, *Ann.*, 1893, **275**, 250.

Cleve, *Bull. soc. chim.*, 1876, **26**, 444.

Vorozhtzov, Gribov, *Chem. Abstracts*, 1933, **27**, 2440.

### 1-Nitronaphthalene-7-sulphonic Acid.

Yellow needles +  $1\frac{1}{2}\text{H}_2\text{O}$  from  $\text{HCl}$ . Aq. M.p.  $135-6^\circ$ .

*NH<sub>4</sub> salt*: yellow plates. Very sol.  $\text{H}_2\text{O}$ .

*K salt*: yellow needles +  $\frac{1}{2}\text{H}_2\text{O}$ . Very sol.  $\text{H}_2\text{O}$ .

*Cu salt*:  $\text{Cu}(\text{C}_{10}\text{H}_6\text{O}_5\text{NS})_2 \cdot 8\text{H}_2\text{O}$ . Green prisms. Loses  $6\text{H}_2\text{O}$  at  $100^\circ$ .

*Ba salt*: needles +  $3\frac{1}{2}\text{H}_2\text{O}$ . Loses  $2\frac{1}{2}\text{H}_2\text{O}$  at  $100^\circ$  and is anhyd. at  $180^\circ$ . Anhyd. salt is sol. 9.1 parts boiling  $\text{H}_2\text{O}$  and 377 parts  $\text{H}_2\text{O}$  at  $17^\circ$ .

*Et ester*: yellow needles from  $\text{EtOH}$ . M.p.  $106^\circ$ .

*Chloride*: plates from  $\text{AcOH}$ . M.p.  $169^\circ$  ( $167^\circ$ ). Spar. sol.  $\text{AcOH}$ ,  $\text{CS}_2$ . Dist. with  $\text{PCl}_5 \rightarrow$  1:7-dichloronaphthalene.

*Amide*: yellow prisms from  $\text{EtOH}$ . M.p.  $223^\circ$ .

*Anilide*: plates from  $\text{EtOH}$ . M.p.  $172-3^\circ$ .

Palmaer, *Ber.*, 1888, **21**, 3260.

Erdmann, Süvern, *Ann.*, 1893, **275**, 251.

Cleve, *Bull. soc. chim.*, 1878, **29**, 414.

Vorozhtzov, Gribov, *Chem. Abstracts*, 1933, **27**, 2440.

Fabrowicz, Lesniański, *Chem. Abstracts*, 1932, **26**, 3791.

### 1-Nitronaphthalene-8-sulphonic Acid.

Needles +  $3\text{H}_2\text{O}$ . M.p.  $115^\circ$  decomp. Very sol.  $\text{H}_2\text{O}$ . Sol.  $\text{EtOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Bitter taste.

*NH<sub>4</sub> salt*: leaflets +  $2\text{H}_2\text{O}$ . Easily sol.  $\text{H}_2\text{O}$ .

*K salt*: needles +  $\text{H}_2\text{O}$ . Very sol.  $\text{H}_2\text{O}$ . Insol.  $\text{KOH}$ . Aq.  $\text{PCl}_5 \rightarrow$  1-chloronaphthalene-8-sulphonyl chloride.

*Ca salt*: leaflets from  $\text{EtOH}$ .

*Me ester*: prisms from  $\text{CHCl}_3$ -pet. ether. M.p.  $124^\circ$ . Sol.  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{CHCl}_3$ . Insol. pet. ether.

*Et ester*: prisms from  $\text{CHCl}_3$ -pet. ether. M.p.  $123.5-124^\circ$  ( $118^\circ$ ). Insol. ligroin.

*Phenyl ester*:  $\text{C}_{16}\text{H}_{11}\text{O}_5\text{NS}$ . MW, 329. M.p.  $132.5-133.5^\circ$ .

*Chloride*: prisms from  $\text{CHCl}_3$ . Decomp. at  $165^\circ$  ( $161^\circ$ ). Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ , pet. ether.

*Amide*: prisms from  $\text{EtOH}$ . M.p.  $190.5-191.5^\circ$ .

*Me-amide*:  $\text{C}_{11}\text{H}_{10}\text{O}_4\text{N}_2\text{S}$ . MW, 266. Cryst. from  $\text{EtOH}$ . M.p.  $195.5-196^\circ$ .

*Di-Me-amide*:  $\text{C}_{12}\text{H}_{12}\text{O}_4\text{N}_2\text{S}$ . MW, 280. Cryst. from  $\text{EtOH}$ . M.p.  $151.5-152.5^\circ$ .

*Et-amide*:  $\text{C}_{12}\text{H}_{12}\text{O}_4\text{N}_2\text{S}$ . MW, 280. Cryst. from  $\text{EtOH}$ . M.p.  $127.5-128.5^\circ$ .

*Di-Et-amide*:  $\text{C}_{14}\text{H}_{16}\text{O}_4\text{N}_2\text{S}$ . MW, 308. Cryst. from  $\text{EtOH}$ . M.p.  $115-16^\circ$ .

*Anilide*: cryst. from  $\text{EtOH}$ . M.p.  $178-178.5^\circ$ .

*Me-anilide*: m.p.  $177.5-178^\circ$ .

*Et-anilide*: m.p.  $170-1^\circ$ .

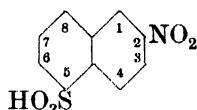
Cleve, *Ber.*, 1890, **23**, 950.

Erdmann, *Ann.*, 1888, **247**, 312.

Erdmann, Süvern, *Ann.*, 1893, **275**, 238.

Steiger, *Helv. Chim. Acta*, 1934, **17**, 701.

Vorozhtzov, Gribov, *Chem. Abstracts*, 1933, **27**, 2440.

**2-Nitronaphthalene-5-sulphonic Acid**

$C_{10}H_7O_5NS$  MW, 253

*Chloride*:  $C_{10}H_6O_4NClS$ . MW, 271.5. Yellowish prisms from  $C_6H_6$ . M.p.  $127^\circ$ .

*Amide*:  $C_{10}H_8O_4N_2S$ . MW, 252. Yellowish plates from EtOH. M.p.  $223-4^\circ$ .

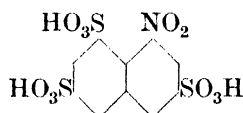
Kappeler, *Ber.*, 1912, 45, 634.

**2-Nitronaphthalene-8-sulphonic Acid.**

*Chloride*: colourless needles from  $C_6H_6$ . M.p.  $169-70^\circ$ .

*Amide*: cryst. from EtOH. M.p.  $261-2^\circ$ .

See previous reference.

**1-Nitronaphthalene-3 : 6 : 8-trisulphonic Acid** (8-Nitronaphthalene-1 : 3 : 6-trisulphonic acid)

$C_{10}H_7O_{11}NS_3$  MW, 413

Hygroscopic needles.

*Na salt*: orange-yellow needles or prisms +  $6H_2O$ .

*Ba salt*: yellow needles +  $8H_2O$ . Efflorescent.

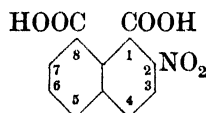
*Pb salt*: orange-yellow needles +  $8H_2O$ . Mod. sol.  $H_2O$ . Efflorescent.

*Aniline salt*: needles +  $2\frac{1}{2}H_2O$ . Sol. EtOH, hot  $H_2O$ . Spar. sol. Et<sub>2</sub>O.

Koch, D.R.P., 56,058.

Kalle, D.R.P., 176,621, (*Chem. Zentr.*, 1906, II, 1746).

Fiery, Schmidt, *Helv. Chim. Acta*, 1921, 4, 386.

**2-Nitronaphthalic Acid** (2-Nitronaphthalene-1 : 8-dicarboxylic acid)

$C_{12}H_7O_6N$  MW, 261

M.p.  $173-5^\circ$ . Heat  $\longrightarrow$  anhydride.

*Anhydride*: 2-nitronaphthalic anhydride.  $C_{12}H_5O_5N$ . MW, 243. M.p.  $190-200^\circ$ .

Morgan, Harrison, *J. Soc. Chem. Ind.*, 1930, 49, 415r.

**3-Nitronaphthalic Acid.**

Known only as its anhydride.

*Anhydride*: 3-nitronaphthalic anhydride. Pale brown leaflets from AcOH. M.p.  $249^\circ$  ( $247^\circ$ ). Insol.  $H_2O$ , EtOH,  $C_6H_6$ .

Graebe, Briones, *Ann.*, 1903, 327, 84.

Anselm, Zuckmayer, *Ber.*, 1899, 32, 3284.

**4-Nitronaphthalic Acid.**

Yellow needles. Decomp. at  $140-50^\circ$ . On heating readily goes to anhydride. Spar. sol. EtOH, Et<sub>2</sub>O, ligroin.

*Di-Et ester*:  $C_{16}H_{15}O_6N$ . MW, 317. Yellow solid. M.p.  $86^\circ$ .

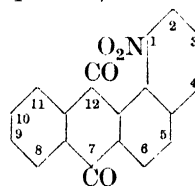
*Anhydride*: 4-nitronaphthalic anhydride. Orange-yellow needles from AcOH. M.p.  $220-2^\circ$ . Sublimes.

*Imide*: 4-nitronaphthalimide.  $C_{12}H_6O_4N_2$ . MW, 242. M.p.  $284^\circ$ .

Quincke, *Ber.*, 1888, 21, 1460.

Dziewoński, *Ber.*, 1903, 36, 3772.

Graebe, Briones, *Ann.*, 1903, 327, 82.

**1-Nitronaphthanthraquinone** (1-Nitro-1' : 2'-benzanthraquinone)

$C_{18}H_9O_4N$  MW, 303

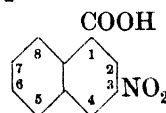
Yellow plates or prisms from  $C_6H_6$ . M.p.  $277-8^\circ$ . Sol.  $PhNO_2$ , boiling AcOH, boiling  $C_6H_6$ . Insol. most cold org. solvents. Sol. conc.  $H_2SO_4$  with orange-red col.

Scholl, *Ber.*, 1911, 44, 2375.

**4-Nitronaphthanthraquinone** (4-Nitro-1' : 2'-benzanthraquinone).

Yellow needles from  $C_6H_6$ . M.p.  $250-1^\circ$ . Sol.  $CHCl_3$ , AcOH,  $C_6H_6$ . Insol. EtOH, Et<sub>2</sub>O. Sol. conc.  $H_2SO_4$  with orange-red col.

See previous reference.

**3-Nitro-1-naphthoic Acid**

$C_{11}H_7O_4N$  MW, 217

Cryst. from EtOH. M.p.  $270.5-271.5^\circ$ .

*Et ester*:  $C_{13}H_{11}O_4N$ . MW, 245. Yellow needles from EtOH or plates from AcOEt. M.p.  $87.5-88.5^\circ$ .

*Amide*:  $C_{11}H_8O_3N_2$ . MW, 216. Cryst. from EtOH. M.p. 280–280.8°. Sol. AcOH. Spar. sol. EtOH, xylene. Insol.  $H_2O$ .

Leuck, Perkins, Whitmore, *J. Am. Chem. Soc.*, 1929, **51**, 1833.

Dziewoński, Kahl, Dymek, *Chem. Zentr.*, 1935, I, 2169.

#### 4-Nitro-1-naphthoic Acid.

Yellowish needles from EtOH. M.p. 220–1°. Sol. EtOH,  $CHCl_3$ , AcOH. Spar. sol.  $C_6H_6$ , ligroin.

*Me ester*:  $C_{12}H_9O_4N$ . MW, 231. M.p. 107.5–108.5°.

*Et ester*: yellow needles. M.p. 57–8° (54°).

*l-Menthyl ester*: cryst. from EtOH. M.p. 63–63.5°.

*Amide*: yellow needles from EtOH. M.p. 218°. Spar. sol. hot  $H_2O$ .

*Nitrile*:  $C_{11}H_6O_2N_2$ . MW, 198. Needles. M.p. 133°. Volatile in superheated steam.

Friedländer, Weisberg, *Ber.*, 1895, **28**, 1841.

Lesser, *Ann.*, 1914, **402**, 16.

Rule, Spence, Bretscher, *J. Chem. Soc.*, 1929, 2520.

Leuck, Perkins, Whitmore, *J. Am. Chem. Soc.*, 1929, **51**, 1835.

#### 5-Nitro-1-naphthoic Acid.

Needles. M.p. 241–2° (239°). Sol. AcOH, warm EtOH. Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Prac. insol. pet. ether. Insol.  $H_2O$ . Sublimes. Ox.  $\rightarrow$  3-nitrophthalic acid. Excess hot  $HNO_3$  (D 1.3)  $\rightarrow$  1:5-dinitronaphthalene.

*Me ester*: yellow needles. M.p. 109–10°.

*Et ester*: needles from EtOH. M.p. 93°.

*Isopropyl ester*:  $C_{14}H_{13}O_4N$ . MW, 259. Cryst. from EtOH. M.p. 101.5°.

*l-Menthyl ester*: cryst. from pet. ether. M.p. 102.5°. Triboluminescent.

*Amide*: cryst. from  $C_6H_6$ . M.p. 235–6°.

*Nitrile*: needles from  $Et_2O$ . M.p. 205°. Very sol.  $CHCl_3$ ,  $C_6H_6$ . Sol. AcOH, hot EtOH. Spar. sol.  $Et_2O$ ,  $CS_2$ , pet. ether. Fuming HCl at 150–60°  $\rightarrow$  5-nitro-1-naphthoic acid.

See last two references above and also Ekstrand, *J. prakt. Chem.*, 1888, **38**, 156, 241, 276.

Graeff, *Ber.*, 1883, **16**, 2249.

#### 6-Nitro-1-naphthoic Acid.

Cryst. from toluene. M.p. 227–227.5°. Sublimes.

*Et ester*: cryst. from EtOH. M.p. 111.5–112°.

*Amide*: needles from EtOH. M.p. 216–5°.

Leuck, Perkins, Whitmore, *J. Am. Chem. Soc.*, 1929, **51**, 1834.

Dziewoński, Kahl, Dymek, *Chem. Zentr.*, 1935, I, 2169.

#### 8-Nitro-1-naphthoic Acid.

Prisms from EtOH. M.p. 215°. Sol. warm AcOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Long heating with  $HNO_3$  (D 1.3)  $\rightarrow$  1:8-dinitronaphthalene.

*Et ester*: yellow cryst. from EtOH. M.p. 68–9°. Very sol. EtOH,  $Et_2O$ .

*l-Menthyl ester*: yellow pyramids from pet. ether. M.p. 122–3°.

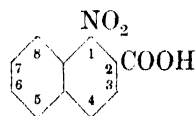
*Amide*: needles from EtOH. M.p. 280°. Very spar. sol. EtOH.

Rule, Barnett, *J. Chem. Soc.*, 1932, 177.

Ekstrand, *J. prakt. Chem.*, 1888, **38**, 156, 276.

Rule, Spence, Bretscher, *J. Chem. Soc.*, 1929, 2521.

#### 1-Nitro-2-naphthoic Acid



$C_{11}H_7O_4N$

MW, 217

Cryst. from AcOH. M.p. 239°.

*Nitrile*:  $C_{11}H_6O_2N_2$ . MW, 198. Brownish cryst. M.p. 138°. Sol. usual org. solvents. Unaffected by prolonged heating with 50%  $H_2SO_4$ . Heat with baryta water  $\rightarrow$  1-hydroxy-2-naphthoic acid.

Friedländer, Littner, *Ber.*, 1915, **48**, 330.

Mayer, Oppenheimer, *Ber.*, 1918, **51**, 1242.

#### 5-Nitro-2-naphthoic Acid.

Yellowish needles from EtOH. M.p. 295° (286–7°). Sol.  $Me_2CO$ . Spar. sol. EtOH,  $Et_2O$ , AcOH,  $CHCl_3$ ,  $C_6H_6$ , pet. ether. Insol.  $H_2O$ .

*Me ester*:  $C_{12}H_9O_4N$ . MW, 231. Yellow needles from EtOH. M.p. 112°.

*Et ester*:  $C_{13}H_{11}O_4N$ . MW, 245. Yellow plates from EtOH. M.p. 111°.

*Isopropyl ester*:  $C_{14}H_{13}O_4N$ . MW, 259. M.p. 75–6°.

*Amide*:  $C_{11}H_8O_3N_2$ . MW, 216. Brownish-yellow needles from  $Me_2CO$ . M.p. 261–3°.

*Nitrile*: cryst. from EtOH. M.p. 172–3°.

(168°). Sublimes in needles. Very sol.  $\text{CHCl}_3$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{EtOH}$ ,  $\text{AcOH}$ . Spar. sol. hot pet. ether. Insol. cold  $\text{H}_2\text{O}$ .

Graeff, *Ber.*, 1883, **16**, 2252.

Friedländer, Heilpern, Spielfogel, *Chem. Zentr.*, 1899, **I**, 288.

Ekstrand, *J. prakt. Chem.*, 1890, **42**, 275.

Cf. Harrison, Royle, *J. Chem. Soc.*, 1926, 84.

### 6-Nitro-2-naphthoic Acid.

Pale yellow plates from  $\text{EtOH}$ . M.p. 310°.

*Et ester*: brownish-yellow plates from  $\text{AcOEt}$ , needles from  $\text{EtOH}$ . M.p. 177°.

Harrison, Royle, *J. Chem. Soc.*, 1926, 89.

### 7-Nitro-2-naphthoic Acid.

Yellow needles from  $\text{EtOH}$ . M.p. 262°. Mod. sol.  $\text{AcOH}$ .

*Et ester*: needles from  $\text{EtOH}$ . M.p. 131°.

See previous reference.

### 8-Nitro-2-naphthoic Acid.

Needles from  $\text{EtOH}$ . M.p. 295° (288°). Sol. 390 parts cold  $\text{EtOH}$ . Sublimes. Alk.  $\text{KMnO}_4 \rightarrow$  trimellitic acid.

*Et ester*: plates from  $\text{C}_6\text{H}_6$  or ligroin. M.p. 121°. Mod. sol.  $\text{EtOH}$ , ligroin.

*Amide*: brownish-yellow needles from  $\text{EtOH}$  or  $\text{Me}_2\text{CO}$ . M.p. 218°.

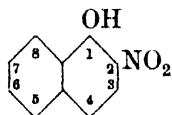
*Nitrile*: brown needles from  $\text{C}_6\text{H}_6$  or  $\text{EtOH}$ . M.p. 143°.

Friedländer, Heilpern, Spielfogel, *Chem. Zentr.*, 1899, **I**, 288.

Ekstrand, *J. prakt. Chem.*, 1890, **42**, 292.

Cf. Harrison, Royle, *J. Chem. Soc.*, 1926, 84.

### 2-Nitro-1-naphthol



$\text{C}_{10}\text{H}_7\text{O}_3\text{N}$

MW, 189

Yellow leaflets from  $\text{EtOH}$ . M.p. 127–8°. Very spar. sol.  $\text{H}_2\text{O}$ . Volatile in steam.

*Me ether*:  $\text{C}_{11}\text{H}_9\text{O}_3\text{N}$ . MW, 203. M.p. 80°.

*Et ether*:  $\text{C}_{12}\text{H}_{11}\text{O}_3\text{N}$ . MW, 217. Yellow needles from ligroin. M.p. 84°. Volatile in steam.

*Acetyl*: m.p. 118°.

Hodgson, Smith, *J. Chem. Soc.*, 1935, 672.  
Vesely, Vojtech, *Chem. Abstracts*, 1929, **23**, 4466.

Hodgson, Kilner, *J. Chem. Soc.*, 1924, **125**, 807.

Charrier, *Gazz. chim. ital.*, 1915, **45**, i, 524.

Grandmougin, Michel, *Ber.*, 1892, **25**, 973.

Heermann, *J. prakt. Chem.*, 1891, **44**, 240.

Pictet, de Krijanowski, *Chem. Zentr.*, 1903, **II**, 1109.

### 3-Nitro-1-naphthol.

Yellow needles. M.p. 167–8°.

Vesely, Dvorak, *Bull. soc. chim.*, 1923, **33**, 329.

### 4-Nitro-1-naphthol.

Needles from  $\text{H}_2\text{O}$ . M.p. 164°. Very sol.  $\text{EtOH}$ ,  $\text{AcOH}$ . Non-volatile in steam. Heat with  $\text{PCl}_5 \rightarrow$  1:4-dichloronaphthalene.

*Me ether*: yellow needles from  $\text{C}_6\text{H}_6$ . M.p. 81° (85–6°).

*Et ether*: needles from  $\text{EtOH}$ . M.p. 120° (116–17°).

*Carbonate*:  $(\text{NO}_2\cdot\text{C}_{10}\text{H}_7\text{O})_2\text{CO}$ . Needles from  $\text{AcOH}$ . M.p. 212°.

*m-Nitrobenzenesulphonyl*: m.p. 135°.

Hodgson, Smith, *J. Chem. Soc.*, 1935, 672.

Bell, *J. Chem. Soc.*, 1933, 287.

Hodgson, Kilner, *J. Chem. Soc.*, 1924, **125**, 807.

Griesheim-Elektron, D.R.P., 117,731, (*Chem. Zentr.*, 1901, **I**, 548).

Reverdin, Kauffmann, *Ber.*, 1895, **28**, 3050.

Deninger, *J. prakt. Chem.*, 1889, **40**, 301.

Francis, *Ber.*, 1906, **39**, 3802.

Woroshzow, *Chem. Zentr.*, 1911, **I**, 651; **II**, 612.

### 5-Nitro-1-naphthol.

Dark yellow cryst. or red needles from  $\text{H}_2\text{O}$ . M.p. 171° (165°). Sol.  $\text{AcOH}$ , toluene. Mod. sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Spar. sol. ligroin. Sol. alkalis with yellowish-brown col.

*Me ether*: yellow needles from pet. ether. M.p. 96–7°.

*Acetyl*: needles from  $\text{EtOH.Aq}$ . M.p. 114°.

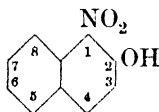
*Benzoyl*: cryst. from  $\text{MeOH}$ . M.p. 109°.

Kauffer, Bräuer, *Ber.*, 1907, **40**, 327.

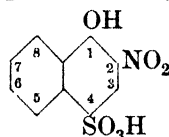
Fichter, Kühnel, *Ber.*, 1909, **42**, 4751.

Vorozhtzov, Kulev, *Chem. Abstracts*, 1929, **23**, 3697.



**8-Nitro-1-naphthol.**Yellow cryst. from  $\text{CHCl}_3$ . M.p.  $130^\circ$ .m-Nitrobenzenesulphonyl: needles from AcOH. M.p.  $166^\circ$ .Bell, *J. Chem. Soc.*, 1933, 287.**1-Nitro-2-naphthol** $\text{C}_{10}\text{H}_7\text{O}_3\text{N}$  MW, 189Yellow needles or leaflets from EtOH. M.p.  $103^\circ$ . Readily sol. EtOH.Me ether:  $\text{C}_{11}\text{H}_9\text{O}_3\text{N}$ . MW, 203. Yellow prisms from AcOH. M.p.  $128^\circ$  ( $126^\circ$ ). Alc.  $\text{NH}_3$  at  $160^\circ \rightarrow$  1-nitro-2-naphthylamine.Et ether:  $\text{C}_{12}\text{H}_{11}\text{O}_3\text{N}$ . MW, 217. Yellow needles from EtOH or AcOH. M.p.  $104-5^\circ$ . Sol.  $\text{Et}_2\text{O}$ . Spar. sol. AcOH, cold EtOH. Insol.  $\text{H}_2\text{O}$ . Volatile in steam.Propylether:  $\text{C}_{13}\text{H}_{13}\text{O}_3\text{N}$ . MW, 231. Yellow needles from EtOH. M.p.  $86^\circ$ .Isopropyl ether: yellow leaflets from EtOH. M.p.  $63^\circ$ .Acetyl: needles from pet. ether. M.p.  $61^\circ$ .m-Nitrobenzenesulphonyl: prisms from AcOH. M.p.  $176^\circ$ .Hartman, Byers, Dickey, *Organic Syntheses*, 1933, XIII, 78.Bell, *J. Chem. Soc.*, 1933, 288.Charrier, *Gazz. chim. ital.*, 1916, **46**, i, 410.Pictet, de Krijanowski, *Chem. Zentr.*, 1903, II, 1109.Liebermann, Jacobson, *Ann.*, 1882, **211**, 46.Francis, *Ber.*, 1906, **39**, 3802.Davis, *Chem. News*, 1896, **74**, 302.**4-Nitro-2-naphthol.**Yellow needles from pet. ether. M.p.  $120^\circ$ . Sol. alkalis with red col.Me ether: brown needles from  $\text{C}_6\text{H}_6$ -EtOH. M.p.  $100-3^\circ$ .m-Nitrobenzenesulphonyl: cryst. from AcOH. M.p.  $149^\circ$ .p-Toluenesulphonyl: pale yellow cryst. from EtOH. M.p.  $122^\circ$ .B.D.C., E.P., 152,437, (*Chem. Abstracts*, 1921, **15**, 761).Morgan, Evens, *J. Chem. Soc.*, 1919, **115**, 1132.Bell, *J. Chem. Soc.*, 1933, 288.Challenor, Ingold, *J. Chem. Soc.*, 1923, **123**, 2080.**5-Nitro-2-naphthol.**Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $147^\circ$ .Et ether: yellow needles from EtOH. M.p.  $115^\circ$ .m-Nitrobenzenesulphonyl: needles from AcOH. M.p.  $166^\circ$ .Friedländer, Szymanski, *Ber.*, 1892, **25**, 2079.Bell, *J. Chem. Soc.*, 1932, 2734.**6-Nitro-2-naphthol.**Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $156-8^\circ$ .Me ether: m.p.  $134^\circ$ .Et ether: needles from  $\text{Et}_2\text{O}$ -pet. ether. M.p.  $114^\circ$ . Very sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ , cold EtOH, pet. ether.  $\text{HNO}_3$  (D 1.14) at  $180-200^\circ \rightarrow$  4-nitrophthalic acid.Gaess, *J. prakt. Chem.*, 1892, **45**, 616; 1892, **46**, 160.Davis, *Chem. News*, 1896, **74**, 302.**8-Nitro-2-naphthol.**Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $144-5^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ .Me ether: m.p.  $69^\circ$ .Et ether: yellow needles from pet. ether. M.p.  $72-3^\circ$ . Very sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Mod. sol. EtOH, pet. ether.Acetyl: needles from EtOH.Aq. M.p.  $101-2^\circ$ .m-Nitrobenzenesulphonyl: cryst. from AcOH. M.p.  $144-6^\circ$ .Bell, *J. Chem. Soc.*, 1932, 2734.

See also previous references.

**2-Nitro-1-naphthol-4-sulphonic Acid** $\text{C}_{10}\text{H}_7\text{O}_6\text{NS}$  MW, 269Heat with HCl under press. at  $150-60^\circ \rightarrow$  2-nitro-1-naphthol.

Mono-K salt: lemon-yellow needles.

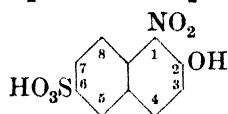
Di-K salt: orange-yellow needles.

Et ether:  $\text{C}_{12}\text{H}_{11}\text{O}_6\text{NS}$ . MW, 297. Hot conc. KOH  $\rightarrow$  2-nitro-1-naphthol-4-sulphonic acid. Fuming HCl at  $150-60^\circ \rightarrow$  2-nitro-1-naphthol. K salt: yellow prisms and plates +  $\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .Witt, Schneider, *Ber.*, 1901, **34**, 3189.**2-Nitro-1-naphthol-7-sulphonic Acid.**

Lemon-yellow needles.

Cu salt: cryst. +  $5\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .Finger, *J. prakt. Chem.*, 1909, **79**, 443.

## 1-Nitro-2-naphthol-6-sulphonic Acid

 $C_{10}H_7O_6NS$ 

MW, 269

*Et ether*: chloride,  $C_{12}H_{10}O_5NClS$ . MW, 315.5. Plates. M.p.  $146^\circ$ . *Amide*:  $C_{12}H_{12}O_5N_2S$ . MW, 296. Prisms or needles. M.p.  $218^\circ$ .

Heermann, *J. prakt. Chem.*, 1894, **49**, 133.Lapworth, *Chem. News*, 1895, **71**, 206.

## 6-Nitro-2-naphthol-4-sulphonic Acid.

Brown cryst. +  $2-5H_2O$  which grinds to intense yellow powder.

Ruggli, Knapp, Merz, Zimmermann, *Helv. Chim. Acta*, 1929, **12**, 1042.

## 6-Nitro-2-naphthol-8-sulphonic Acid.

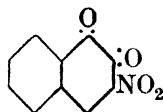
Yellow prisms +  $4H_2O$  from  $H_2O$ . At  $150^\circ$  loses  $2H_2O$ . Mod. sol. cold  $H_2O$ . Spar. sol. EtOH. Insol. Et<sub>2</sub>O.

*K salt*: orange prisms.

*Ba salt*: dark yellow prisms +  $6\frac{1}{2}H_2O$ .

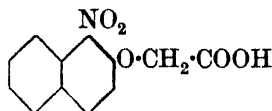
Jacchia, *Ann.*, 1902, **323**, 122.

## 3-Nitro-1 : 2-naphthoquinone

 $C_{10}H_5O_4N$ 

MW, 203

Red cryst. from AcOH. M.p.  $158^\circ$ . Very sol. hot AcOH. Sol.  $C_6H_6$ , boiling EtOH. Spar. sol. Et<sub>2</sub>O. Insol.  $CS_2$ . Hot sulphurous acid  $\rightarrow$  3-nitro-1 : 2-dihydroxynaphthalene. Phenylhydrazine  $\rightarrow$  3-nitro-1 : 2-dihydroxynaphthalene.

Zincke *et al.*, *Ann.*, 1892, **268**, 273, 297.Zincke, Noack, *Ann.*, 1897, **295**, 12, (Footnote).Zärtling, *Ber.*, 1890, **23**, 175.1-Nitro- $\beta$ -naphthoxyacetic Acid $C_{12}H_9O_5N$ 

MW, 247

Yellow prisms from AcOH. M.p.  $192^\circ$  ( $188-9^\circ$ ). Very sol. hot EtOH, hot AcOH. Sol. Et<sub>2</sub>O,  $C_6H_6$ . Very spar. sol.  $H_2O$ .

*Et ester*:  $C_{14}H_{13}O_5N$ . MW, 275. Yellowish needles. M.p.  $100^\circ$ .

Dict. of Org. Comp.—III.

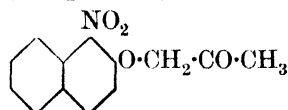
*Chloride*:  $C_{12}H_8O_4NCl$ . MW, 265.5. Yellow leaflets. M.p.  $94^\circ$ . Sol.  $C_6H_6$ . Insol. ligroin.

*Amide*:  $C_{12}H_{10}O_4N_2$ . MW, 246. Yellow needles from EtOH. M.p.  $189^\circ$ . Sol. Et<sub>2</sub>O, hot EtOH. Spar. sol. hot  $H_2O$ .

*Anilide*: yellow needles. M.p.  $139^\circ$ .

Spitzer, *Ber.*, 1901, **34**, 3193.Lees, Shedden, *J. Chem. Soc.*, 1903, **83**, 758

Badische, D.R.P., 58,614.

1-Nitro- $\beta$ -naphthoxyacetone $C_{13}H_{11}O_4N$ 

MW, 245

Yellowish needles. M.p.  $145^\circ$ . Readily sol. hot EtOH. Mod. sol. Et<sub>2</sub>O, AcOH. Insol.  $H_2O$ .

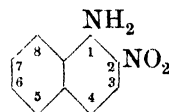
*Oxime*: m.p.  $158^\circ$ .

*Semicarbazone*: yellowish needles. M.p.  $208^\circ$ .

*Phenylhydrazone*: yellow plates. M.p.  $120^\circ$ .

Stoermer, Franke, *Ber.*, 1898, **31**, 759.

## 2-Nitro-1-naphthylamine

 $C_{10}H_8O_2N_2$ 

MW, 188

Reddish-yellow prisms from EtOH. M.p.  $144^\circ$ . Hot alkali  $\rightarrow$  2-nitro-1-naphthol.

*N-Me*: see 2-Nitro-*N*-methyl-1-naphthylamine.

*N-Et*:  $C_{12}H_{12}O_2N_2$ . MW, 216. Red needles. M.p.  $77^\circ$ .

*N-Phenyl*: orange cryst. from EtOH. M.p.  $110-11^\circ$ .

*N-Acetyl*: 2-nitro-1-acetnaphthalide. Yellow needles from EtOH or AcOH. M.p.  $199^\circ$ . Very stable towards  $HNO_2$ .

*N-Diacetyl*: yellow prisms from AcOH. M.p.  $115^\circ$ .

*N-Benzoyl*: yellowish prisms from EtOH-AcOH. M.p.  $175^\circ$ . Very sol. EtOH,  $CHCl_3$ , AcOH,  $C_6H_6$ .

Meisenheimer, Patzig, *Ber.*, 1906, **39**, 2541.Lellmann, *Ber.*, 1887, **20**, 893.Lellmann, Remy, *Ber.*, 1886, **19**, 797.Hodgson, Walker, *J. Chem. Soc.*, 1933, 1205.Hoogeveen, *Rec. trav. chim.*, 1931, **50**, 39.Bamberger, *Ber.*, 1922, **55**, 3389.

**3-Nitro-1-naphthylamine.**

M.p. 136-7°.

*Acetyl*: 3-nitro-1-acetnaphthalide. M.p. 255°.Veselý, Dvorák, *Bull. soc. chim.*, 1923, **33**, 328.**4-Nitro-1-naphthylamine.**Orange needles from EtOH. M.p. 195° (191°). Mod. sol. EtOH, AcOH. Hot KOH  $\rightarrow$  NH<sub>3</sub> + 4-nitro-1-naphthol.*N-Me*: see 4-Nitro-*N*-methyl-1-naphthylamine.*N-Et*: orange cryst. from AcOH. M.p. 179-80° (176-7°). *N-Acetyl*: needles from EtOH. Aq. M.p. 112-13°. *N-Benzoyl*: yellowish prisms from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 121°.*N-Benzyl*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 156°.*N-Acetyl*: 4-nitro-1-acetnaphthalide. Needles. M.p. 190°.*N-Diacetyl*: yellow needles from EtOH. M.p. 144°.*N-Benzoyl*: prisms from AcOH. M.p. 224°.*N-Benzenesulphonyl*: m.p. 173°.*N-p-Toluenesulphonyl*: yellow needles. M.p. 185°.Meldola, Streatfield, *J. Chem. Soc.*, 1893, **63**, 1055.Lellmann, Remy, *Ber.*, 1886, **19**, 797.Morgan, Couzens, *J. Chem. Soc.*, 1910, **97**, 1693.Hodgson, Walker, *J. Chem. Soc.*, 1933, 1205.Chem. Farbr. Griesheim-Elektron, D.R.P., 117,006, (*Chem. Zentr.*, 1901, I, 237).**5-Nitro-1-naphthylamine.**Red needles from H<sub>2</sub>O. M.p. 118-19°.*N-Formyl*: yellow needles. M.p. 199°.*N-Acetyl*: 5-nitro-1-acetnaphthalide. Brownish prisms from AcOH. M.p. 220°.*N-Benzenesulphonyl*: needles from EtOH. Aq. M.p. 183°.Badische, D.R.P., 145,191, (*Chem. Zentr.*, 1903, II, 1097).Morgan, Jones, *J. Soc. Chem. Ind.*, 1923, **42**, 341r.Morgan, Micklethwait, *J. Chem. Soc.*, 1906, **89**, 7.Hodgson, Walker, *J. Chem. Soc.*, 1933, 1346.Vorohtzov, Kulev, *Chem. Abstracts*, 1929, **23**, 3697.Hodgson, E.P., 392,914, (*Chem. Abstracts*, 1933, **27**, 5757).**6-Nitro-1-naphthylamine.**

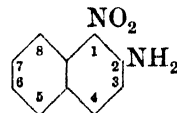
M.p. 143°.

*N-Acetyl*: 6-nitro-1-acetnaphthalide. M.p. 232-3°.Veselý, Dvorák, *Bull. soc. chim.*, 1923, **33**, 330.**7-Nitro-1-naphthylamine.**

Red needles from AcOH. M.p. 133-4° (122-3°).

*N-Acetyl*: 7-nitro-1-acetnaphthalide. M.p. 206-7°.Schroeter, D.R.P., 563,627, (*Chem. Abstracts*, 1933, **27**, 995).Schroeter *et al.*, *Ber.*, 1930, **63**, 1317.Veselý, Dvorák, *Bull. soc. chim.*, 1923, **33**, 330.**8-Nitro-1-naphthylamine.**

Red cryst. from pet. ether. M.p. 96-7°.

*N-Me*: see 8-Nitro-*N*-methyl-1-naphthylamine.*N-Acetyl*: 8-nitro-1-acetnaphthalide. Needles from H<sub>2</sub>O. M.p. 191°.*N-Benzenesulphonyl*: needles from EtOH. M.p. 194°.Morgan, Micklethwait, *J. Chem. Soc.*, 1906, **89**, 7.Meldola, Streatfield, *J. Chem. Soc.*, 1893, **63**, 1055.Morgan, Jones, *J. Soc. Chem. Ind.*, 1923, **42**, 341r.**1-Nitro-2-naphthylamine**C<sub>10</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub>

MW, 188

Orange-yellow needles from EtOH. M.p. 126-7° (123-4°). Sol. EtOH, Me<sub>2</sub>CO, AcOH. Mod. sol. boiling H<sub>2</sub>O.*N-Me*: see 1-Nitro-*N*-methyl-2-naphthylamine.*N-Et*: C<sub>12</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 216. Orange-red prisms from EtOH. M.p. 100-1°. *N-p-Toluenesulphonyl*: needles from EtOH. M.p. 152-3°.*N-Nitroso*: cryst. from EtOH. M.p. 90°.*N-Phenyl*: red prisms from EtOH. M.p. 110-11°.*N-Acetyl*: 1-nitro-2-acetnaphthalide. Yellow cryst. from EtOH. M.p. 123.5°. Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Mod. sol. hot H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O, ligroin.*N-Benzoyl*: cryst. + 1 mol. C<sub>6</sub>H<sub>6</sub> from C<sub>6</sub>H<sub>6</sub>. M.p. (benzene-free), 168°.

*N*-Benzenesulphonyl : yellowish prisms. M.p. 156°.

*N*-p-Toluenesulphonyl : orange-yellow prisms from EtOH. M.p. 160-1°.

*B*, *C*<sub>6</sub>*H*<sub>3</sub>(*NO*<sub>2</sub>)<sub>3</sub>-1 : 3 : 5 : yellow needles from EtOH. M.p. 115.5-116°.

Hartman, Smith, *Organic Syntheses*, 1933, XIII, 72.

Liebermann, Jacobson, *Ann.*, 1882, 211, 64.

Meldola, Lane, *J. Chem. Soc.*, 1904, 85, 1603.

Morgan, Micklethwait, *J. Chem. Soc.*, 1912, 101, 148.

Veselý, Dvorák, *Bull. soc. chim.*, 1923, 33, 331.

#### 5-Nitro-2-naphthylamine.

Red needles from EtOH. M.p. 143.5°. Sol. AcOH, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Insol. ligroin.

*N*-Acetyl : 5-nitro-2-acetnaphthalide. Yellow needles from C<sub>6</sub>H<sub>6</sub>. M.p. 186°.

*N*-Benzoyl : needles. M.p. 181.5°.

Friedländer, Szymanski, *Ber.*, 1892, 25, 2077.

Hirsch, D.R.P., 57,491.

Veselý, Dvorák, *Bull. soc. chim.*, 1923, 33, 327.

Morgan, Chazan, *J. Soc. Chem. Ind.*, 1922, 41, 1r.

#### 8-Nitro-2-naphthylamine.

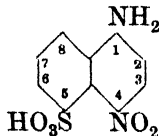
Red needles. M.p. 104-5° (103.5°). Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

*N*-Acetyl : 8-nitro-2-acetnaphthalide. Yellow needles from EtOH. M.p. 195.5°.

*N*-Benzoyl : greenish-yellow needles from EtOH. M.p. 162°.

See first three references above and also Morgan, Gilmour, *J. Soc. Chem. Ind.*, 1922, 41, 61r.

#### 4-Nitro-1-naphthylamine-5-sulphonic Acid



C<sub>10</sub>H<sub>8</sub>O<sub>5</sub>N<sub>2</sub>S

MW, 268

Needles. Spar. sol. H<sub>2</sub>O.

Bayer, D.R.P., 133,951, (*Chem. Zentr.*, 1902, II, 867).

#### 4-Nitro-1-naphthylamine-6-sulphonic Acid.

Yellow cryst. from H<sub>2</sub>O.

Cassella, D.R.P., 73,502.

Bayer, D.R.P., 228,764, (*Chem. Zentr.*, 1911, I, 105).

#### 4-Nitro-1-naphthylamine-7-sulphonic Acid.

Reddish-brown amorph. powder from H<sub>2</sub>O. Mod. sol. hot H<sub>2</sub>O.

See first reference above.

#### 5-Nitro-1-naphthylamine-2-sulphonic Acid.

Yellow flakes. Spar. sol. H<sub>2</sub>O.

*Na* salt : yellow leaflets.

Cassella, D.R.P., 70,890.

#### 5-Nitro-1-naphthylamine-4-sulphonic Acid (8-Nitronaphthionic acid).

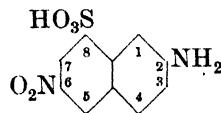
Needles.

*N*-Acetyl : greenish-yellow cryst. Hot dil. H<sub>2</sub>SO<sub>4</sub> → 5-nitro-1-naphthylamine. *NH*<sub>4</sub> salt : yellow needles from H<sub>2</sub>O.

Nietzki, Zúbelen, *Ber.*, 1889, 22, 452.

Bucherer, Uhlmann, *J. prakt. Chem.*, 1909, 80, 221.

#### 6-Nitro-2-naphthylamine-8-sulphonic Acid



C<sub>10</sub>H<sub>8</sub>O<sub>5</sub>N<sub>2</sub>S

MW, 268

Yellowish amorph. powder.

*NH*<sub>4</sub> salt : dark red prisms.

*Ba* salt : red cryst. + 4½ H<sub>2</sub>O

Jacchia, *Ann.*, 1902, 323, 119.

Friedländer, Lucht, *Ber.*, 1893, 26, 3033.

#### 8-Nitro-2-naphthylamine-6-sulphonic Acid.

Grey powder. Prac. insol. H<sub>2</sub>O.

*Na* salt : orange.

*NH*<sub>4</sub> salt : orange.

Voroshcov, Gribov, *Chem. Abstracts*, 1924, 18, 1124.

#### 1-Nitro-octane

CH<sub>3</sub>·[CH<sub>2</sub>]<sub>6</sub>·CH<sub>2</sub>NO<sub>2</sub>  
C<sub>8</sub>H<sub>17</sub>O<sub>2</sub>N

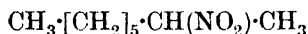
MW, 159

Yellow liq. B.p. 206–10° part decomp.  
D<sub>20</sub> 0.9346.

Worstell, *Am. Chem. J.*, 1898, 20, 213;  
1899, 21, 228.

Eichler, *Ber.*, 1879, 12, 1883.

### 2-Nitro-octane



C<sub>8</sub>H<sub>17</sub>O<sub>2</sub>N MW, 159

*d.*

Pale yellow oil. B.p. 102–5°/23 mm. D<sub>20</sub>  
0.9224.  $n_D^{20}$  1.4324.  $[\alpha]_D^{25} + 15.84^\circ$  in EtOH.

*Na salt*:  $[\alpha]_D^{25} + 3.31^\circ$  in EtOH.

*K salt*:  $[\alpha]_D^{25} + 3.74$  in EtOH.

*l.*

B.p. 100–3°/18 mm. D<sub>20</sub> 0.9165.  $n_D^{20}$  1.4292.  
 $[\alpha]_D^{25} - 10.8^\circ$  in EtOH.

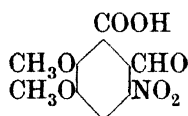
*dl.*

B.p. 210–12° decomp., 123–4°/40 mm. D°  
0.93645.

Shriver, Young, *J. Am. Chem. Soc.*, 1930,  
52, 3337.

Konowalow, *Chem. Zentr.*, 1899, I, 1063.

### 3-Nitro-opianic Acid



C<sub>10</sub>H<sub>9</sub>O<sub>7</sub>N MW, 255

Yellow prisms from H<sub>2</sub>O. M.p. 169–70°.  
Spar. sol. H<sub>2</sub>O.  $k = 2.91 \times 10^{-6}$  at 25°.  
KMnO<sub>4</sub> in hot carbonate sol. → 6-nitro-  
hemipinic acid.

*Me ester*: C<sub>11</sub>H<sub>11</sub>O<sub>7</sub>N. MW, 269. Needles  
from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 78°. KMnO<sub>4</sub> →  
6-nitrohemipinic acid. *Diacetate*: cryst. from  
C<sub>6</sub>H<sub>6</sub>. M.p. 159–60°.

*ψ-Me ester*: 4-nitro-3 : 6 : 7-trimethoxy-  
phthalide. Needles from MeOH. M.p. 181.5–  
182.5°. Sol. Me<sub>2</sub>CO, hot C<sub>6</sub>H<sub>6</sub>. Stable to  
KMnO<sub>4</sub>.

*Et ester*: C<sub>12</sub>H<sub>13</sub>O<sub>7</sub>N. MW, 283. Yellow  
needles and plates from C<sub>6</sub>H<sub>6</sub>. M.p. 80–1°.  
*Diacetate*: cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p.  
100°.

*ψ-Et ester*: 4-nitro-6 : 7-dimethoxy-3-ethoxy-  
phthalide. Needles from CS<sub>2</sub>. M.p. 96°. Very  
sol. Et<sub>2</sub>O. Sol. CS<sub>2</sub>, hot C<sub>6</sub>H<sub>6</sub>. Hyd. by H<sub>2</sub>O.

*Chloride*: C<sub>10</sub>H<sub>8</sub>O<sub>6</sub>NCl. MW, 273.5. Yel-  
lowish leaflets from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 137–8°.  
Sol. C<sub>6</sub>H<sub>6</sub>, hot CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O, ligroin.

*Amide*: C<sub>10</sub>H<sub>10</sub>O<sub>6</sub>N<sub>2</sub>. MW, 254. Straw-  
yellow needles from H<sub>2</sub>O. M.p. 203° decomp.  
Sol. AcOH, hot H<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>.

*Anhydride*: C<sub>20</sub>H<sub>16</sub>O<sub>13</sub>N<sub>2</sub>. MW, 492. Cryst.  
from AcOEt. M.p. 250°.

Prinz, *J. prakt. Chem.*, 1881, 24, 357.

Claus, Predari, *J. prakt. Chem.*, 1897, 55,  
173, (*Footnote*).

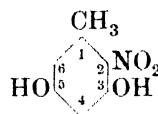
Wegscheider, Müller, Chiari, *Monatsh.*,  
1908, 29, 742.

Wegscheider, Kusy, v. Dúbrav, v.  
Rušnov, *Monatsh.*, 1903, 24, 801.

Wegscheider, Späth, *Monatsh.*, 1916, 37,  
299.

Wegscheider, Müller, *Ann.*, 1923, 433,  
33.

### 2-Nitro-orscinol (2-Nitro-3 : 5-dihydroxy- toluene)



C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>N MW, 169

Yellow needles + H<sub>2</sub>O from H<sub>2</sub>O. M.p.  
122°. Very sol. EtOH, Et<sub>2</sub>O. Sol. C<sub>6</sub>H<sub>6</sub>,  
CHCl<sub>3</sub>, ligroin. Non-volatile in steam.

*3-Me ether*: C<sub>8</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 183. Brownish-  
yellow cryst. from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 129–31°.  
Sol. EtOH, Et<sub>2</sub>O, AcOH. Insol. CS<sub>2</sub>, ligroin.  
Non-volatile in steam.

*5-Me ether*: yellow needles from EtOH or  
ligroin. M.p. 104–6°. Very sol. hot EtOH,  
C<sub>6</sub>H<sub>6</sub>, AcOEt. Sol. Et<sub>2</sub>O, AcOH. Volatile in  
steam.

*3-Et ether*: C<sub>9</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 197. Yellow  
needles. M.p. 103°. Non-volatile in steam.

*5-Et ether*: yellow needles. M.p. 54°.  
Volatile in steam.

Henrich, Meyer, *Ber.*, 1903, 36, 886.

Henrich, Nachtigall, *ibid.*, 893.

Weselsky, Benedikt, *Monatsh.*, 1881, 2,  
370.

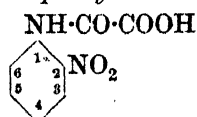
### 4-Nitro-orscinol (4-Nitro-3 : 5-dihydroxy- toluene).

Orange needles from EtOH. M.p. 127°. Sol.  
EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOH. Mod. sol.  
hot ligroin. Spar. sol. H<sub>2</sub>O. Volatile in steam.  
Sublimes.

Henrich, Meyer, *Ber.*, 1903, 36, 886.

Weselsky, *Ber.*, 1874, 7, 439.

### 2-Nitro-oxanilic Acid (Oxalic acid mono-o- nitroanilide, o-nitrophenyloxamic acid)



C<sub>8</sub>H<sub>6</sub>O<sub>5</sub>N<sub>2</sub>

MW, 210

Golden needles from  $\text{H}_2\text{O}$ . M.p.  $112^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH, AcOH. Spar. sol.  $\text{Et}_2\text{O}$ .

*Et ester*:  $\text{C}_{10}\text{H}_{10}\text{O}_5\text{N}_2$ . MW, 238. Yellow needles from EtOH or AcOH. M.p.  $113^\circ$  ( $108^\circ$ ).

*o*-Nitroanilide: see under Oxalic Acid.

Aschan, *Ber.*, 1885, **18**, 2937.

Abderhalden, Ehrenwall, Schwab, Zumstein, *Chem. Abstracts*, 1933, **27**, 107.

Kuhlmann, F.P., 649,328, (*Chem. Abstracts*, 1929, **23**, 2988).

Pickard, Allen, Bowdler, Carter, *J. Chem. Soc.*, 1902, **81**, 1568.

**3-Nitro-oxanilic Acid** (*Oxalic acid mono-m-nitroanilide, m-nitrophenyloxamic acid*).

M.p.  $158^\circ$ .

*Et ester*: yellow needles from EtOH. M.p.  $150^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ .

*Amide*:  $\text{C}_8\text{H}_7\text{O}_4\text{N}_3$ . MW, 209. Needles from AcOH. M.p.  $268-9^\circ$ .

*Anilide*: yellow needles from  $\text{PhNO}_2$ . M.p.  $204^\circ$ .

*m*-Nitroanilide: see under Oxalic Acid.

Anselmino, *Chem. Zentr.*, 1906, **I**, 753.

Kuhlmann, F.P., 649,328, (*Chem. Abstracts*, 1929, **23**, 2988).

Pickard, Allen, Bowdler, Carter, *J. Chem. Soc.*, 1902, **81**, 1569.

Jacobs, Heidelberger, *J. Am. Chem. Soc.*, 1917, **39**, 1452.

**4-Nitro-oxanilic Acid** (*Oxalic acid mono-p-nitroanilide, p-nitrophenyloxamic acid*).

Yellowish needles +  $1\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $216^\circ$ . Mod. sol. EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_9\text{H}_8\text{O}_5\text{N}_2$ . MW, 224. Yellow cryst. from MeOH. M.p.  $232^\circ$ .

*Et ester*: needles from AcOH. M.p.  $172^\circ$  ( $166^\circ$ ). Very sol.  $\text{CHCl}_3$ , AcOH, AcOEt, Py. Sol.  $\text{Et}_2\text{O}$ , EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Very spar. sol.  $\text{H}_2\text{O}$ , pet. ether.

*Amide*: m.p.  $308-10^\circ$  decomp. Sol. boiling AcOH, AcOEt. Spar. sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

*Anilide*: grey needles from AcOEt. M.p.  $251-2^\circ$ .

*p*-Nitroanilide: see under Oxalic Acid.

Mumm, Hesse, Volquartz, *Ber.*, 1915, **48**, 391.

Suida, *Monatsh.*, 1910, **31**, 605.

Pickard, Allen, Bowdler, Carter, *J. Chem. Soc.*, 1902, **81**, 1570.

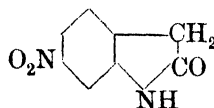
Schultz, Rohde, Herzog, *J. prakt. Chem.*, 1906, **74**, 82.

Aschan, *Ber.*, 1885, **18**, 2936.

Tierie, *Rec. trav. chim.*, 1933, **52**, 420.

Kuhlmann, F.P., 649,328, (*Chem. Abstracts*, 1929, **23**, 2988).

### 6-Nitro-oxindole



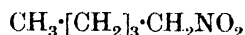
$\text{C}_8\text{H}_6\text{O}_3\text{N}_2$

MW, 178

Yellow needles from  $\text{H}_2\text{O}$  or EtOH. Decomp. at  $175^\circ \rightarrow$  a colourless sublimate. Sol. alkalis with reddish-yellow col.

Baeyer, *Ber.*, 1879, **12**, 1313.

### 1-Nitropentane



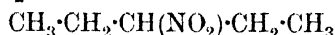
$\text{C}_5\text{H}_{11}\text{O}_2\text{N}$

MW, 117

Liq. with rancid odour. B.p.  $172-3^\circ/760$  mm.,  $88-90^\circ/64$  mm.  $D^{20}$  0.9475.  $n_D$  1.4218. Sweet taste.

Henry, *Rec. trav. chim.*, 1905, **24**, 352.

### 3-Nitropentane



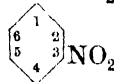
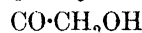
$\text{C}_5\text{H}_{11}\text{O}_2\text{N}$

MW, 117

Liq. B.p.  $152-5^\circ/746$  mm.  $D^\circ$  0.9575.

Bowad, *J. prakt. Chem.*, 1893, **48**, 380.

***m*-Nitrophenacyl Alcohol** (*3-Nitrobenzoyl-carbinol, 3-nitro- $\omega$ -hydroxyacetophenone*)



$\text{C}_8\text{H}_7\text{O}_4\text{N}$

MW, 181

Pale yellow cryst. M.p.  $92.5-93^\circ$ . Aq. sol. reduces  $\text{NH}_3 \cdot \text{AgNO}_3$  and Fehling's. Alk.  $\text{Cu}(\text{OH})_2 \rightarrow$  *m*-nitromandelic acid.  $\text{KMnO}_4 \rightarrow$  *m*-nitrobenzoic acid.

*Acetyl*: cryst. from  $\text{Et}_2\text{O}$ -ligroin. M.p.  $53^\circ$ .

*Semicarbazone*: cryst. from EtOH. M.p.  $214^\circ$ .

Evans, Brooks, *J. Am. Chem. Soc.*, 1908, **30**, 407.

Baker, *J. Chem. Soc.*, 1931, 2422.

***p*-Nitrophenacyl Alcohol** (*4-Nitrobenzoyl-carbinol, 4-nitro- $\omega$ -hydroxyacetophenone*).

M.p.  $121^\circ$ . Sol. hot alkalis.

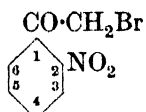
*Acetyl*: cryst. from AcOEt-ligroin. M.p.  $124^\circ$ .

*Phenylhydrazone*: needles from  $\text{C}_6\text{H}_6$ . M.p.  $178^\circ$ .

Baker, *J. Chem. Soc.*, 1931, 2426.

Engler, Zielke, *Ber.*, 1889, **22**, 204.

**o-Nitrophenacyl bromide** ( $\omega$ -Bromo-2-nitroacetophenone)



$\text{C}_8\text{H}_6\text{O}_3\text{NBr}$

MW, 244

Needles from ligroin. M.p.  $55-6^\circ$ . Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. ligroin. Alcoholic (NH<sub>4</sub>)<sub>2</sub>S  $\longrightarrow$  indigo.

Gevekoht, *Ann.*, 1883, 221, 327.

Arndt, Eistert, Partale, *Ber.*, 1927, 60, 1369.

**m-Nitrophenacyl bromide** ( $\omega$ -Bromo-3-nitroacetophenone).

Needles from EtOH.Aq. M.p.  $96^\circ$ . Sol. EtOH, CHCl<sub>3</sub>, CS<sub>2</sub>. Very spar. sol. Et<sub>2</sub>O. KMnO<sub>4</sub>  $\longrightarrow$  m-nitrobenzoic acid.

Oxime: yellowish needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p.  $126.5-127^\circ$ . Acetyl: yellowish needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p.  $64-5^\circ$ .

Korten, Scholl, *Ber.*, 1901, 34, 1909.

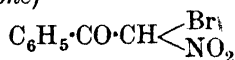
Evans, Brooks, *J. Am. Chem. Soc.*, 1908, 30, 406.

**p-Nitrophenacyl bromide** ( $\omega$ -Bromo-4-nitroacetophenone).

Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p.  $98^\circ$ . Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, hot EtOH.

Baker, *J. Chem. Soc.*, 1931, 2420.

**$\omega$ -Nitrophenacyl bromide** ( $\omega$ -Bromo- $\omega$ -nitroacetophenone)



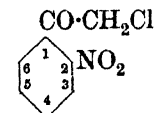
$\text{C}_8\text{H}_6\text{O}_3\text{NBr}$

MW, 244

Cryst. from Et<sub>2</sub>O. M.p.  $61.5^\circ$ . Sol. EtOH and warm aq. Na<sub>2</sub>CO<sub>3</sub>. Alc. KOH  $\longrightarrow$  intense yellow K salt.

Thiele, Haeeckel, *Ann.*, 1902, 325, 13.

**o-Nitrophenacyl chloride** ( $\omega$ -Chloro-2-nitroacetophenone)



$\text{C}_8\text{H}_6\text{O}_3\text{NCl}$

MW, 199.5

Needles from ligroin or MeOH. M.p.  $66-7^\circ$ . Very sol. Et<sub>2</sub>O. Sol. EtOH.

Arndt, Eistert, Partale, *Ber.*, 1927, 60, 1369.

**m-Nitrophenacyl chloride** ( $\omega$ -Chloro-3-nitroacetophenone).

Prisms from Et<sub>2</sub>O. M.p.  $103^\circ$ .

Baker, *J. Chem. Soc.*, 1931, 2420.

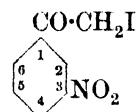
Barkenbus, Clements, *J. Am. Chem. Soc.*, 1934, 56, 1369.

**p-Nitrophenacyl chloride** ( $\omega$ -Chloro-4-nitroacetophenone).

Brown needles from EtOH. M.p.  $107^\circ$ .

Dale, Nierenstein, *Ber.*, 1927, 60, 1027.

**m-Nitrophenacyl iodide** ( $\omega$ -Iodo-3-nitroacetophenone)



$\text{C}_8\text{H}_6\text{O}_3\text{NI}$

MW, 291

Prisms from Et<sub>2</sub>O-ligroin. M.p.  $96^\circ$ .

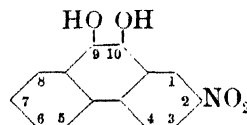
Baker, *J. Chem. Soc.*, 1931, 2420.

**p-Nitrophenacyl iodide** ( $\omega$ -Iodo-4-nitroacetophenone).

Needles from AcOEt-ligroin. M.p.  $97-8^\circ$ .

See previous reference.

**2-Nitrophenanthrahydroquinone** (2-Nitro-9:10-dihydroxyphenanthrene)



$\text{C}_{14}\text{H}_9\text{O}_4\text{N}$

MW, 255

Yellowish-brown cryst. M.p. about  $220^\circ$  decomp. Dil. NaOH  $\longrightarrow$  brownish-violet sol. Conc. H<sub>2</sub>SO<sub>4</sub>  $\longrightarrow$  reddish-violet sol.

Di-Me ether: C<sub>16</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 283. Yellow needles from EtOH. M.p.  $126-7^\circ$ . Sol. EtOH, Me<sub>2</sub>CO, AcOH.

Diacyl: pale yellow needles. M.p.  $258^\circ$ .

Schmidt, Austin, *Ber.*, 1903, 36, 3732.

Jakubowitsch, Worobjowa, *J. prakt. Chem.*, 1935, 143, 285.

**3-Nitrophenanthrahydroquinone** (3-Nitro-9:10-dihydroxyphenanthrene).

Red needles with blue reflex from 60% AcOH. M.p.  $222-3^\circ$ . Sol. EtOH, MeOH, Et<sub>2</sub>O, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. Very dil. NaOH  $\longrightarrow$  blue sol. which absorbs oxygen. Reduces AgNO<sub>3</sub> and FeCl<sub>3</sub>. Conc. H<sub>2</sub>SO<sub>4</sub>  $\longrightarrow$  red sol.  $\longrightarrow$  pale yellow on dilution.

Acetyl: yellow needles from Ac<sub>2</sub>O. M.p.

234–5° decomp. NaOH → deep red sol. → blue on standing.

Schmidt, Kämpf, *Ber.*, 1902, **35**, 3125.

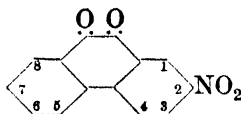
Schmidt, *ibid.*, 3132.

**4-Nitrophenanthrahydroquinone (4-Nitro-9:10-dihydroxyphenanthrene).**

*Diacetyl*: needles from 60% AcOH. M.p. 222–3° decomp.

Schmidt, Kämpf, *Ber.*, 1903, **36**, 3737.

**2-Nitrophenanthraquinone**



$C_{14}H_7O_4N$

MW, 253

Golden-yellow leaflets from AcOH. M.p. 260° (257°). Spar. sol. AcOH. Insol. EtOH. Sn + HCl → 2-aminophenanthraquinone.

*Oxime*: yellowish-green needles. M.p. 213° decomp.

*Thiosemicarbazone*: red cryst. from Py. M.p. 234–5°.

Schmidt, Heinle, *Ber.*, 1911, **44**, 1497.

Werner, *Ber.*, 1904, **37**, 3086.

Schmidt, Austin, *Ber.*, 1903, **36**, 3731.

**3-Nitrophenanthraquinone.**

Orange needles from AcOH. M.p. 281–2° (275°). Spar. sol. org. solvents. Conc.  $H_2SO_4$  → red col. KOH.Aq. → dark green col.

*Imide*: green cryst. from  $C_6H_6$ . Decomp. at 203°. Sol.  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

*Oxime*: yellow needles from  $C_6H_6$ . M.p. 240°. Mod. sol. hot  $C_6H_6$ . Spar. sol. other solvents. Alc.  $FeCl_3$  → blood-red col. *Semicarbazone*: greenish-yellow cryst. from EtOH. M.p. 249–50° decomp.

*Dioxime*: yellowish-red needles from EtOH. M.p. 200° decomp. Sol.  $CS_2$ ,  $C_6H_6$ . Spar. sol. EtOH. *Di-Me*: pale yellow needles from EtOH. M.p. 190–2°. Very sol.  $CHCl_3$ . Sol.  $C_6H_6$ . Spar. sol. EtOH. *Diacetyl*: pale yellow plates from  $C_6H_6$ . M.p. 183° decomp. Sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH.

*Semicarbazone*: m.p. 254° decomp.

Schmidt, Söll, *Ber.*, 1908, **41**, 3683.

Schmidt, Kämpf, *Ber.*, 1902, **35**, 3120.

Schmidt, Schärer, Glatz, *Ber.*, 1911, **44**, 282.

**4-Nitrophenanthraquinone.**

Pale yellow needles from EtOH. M.p. 179–80°. Very sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ , AcOH,

AcOEt,  $C_6H_6$ . Spar. sol.  $Et_2O$ ,  $CS_2$ . Sol. dil. alkalis. Conc.  $H_2SO_4$  → dark red sol. → light brown on dilution.

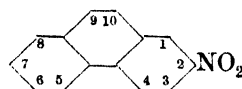
*Oxime*: greenish-yellow needles from EtOH. M.p. 169–70°. Mod. sol. EtOH,  $Me_2CO$ , AcOEt,  $C_6H_6$ . Alc.  $FeCl_3$  → deep brownish-red col.

*Thiosemicarbazone*: red cryst. from EtOH–Py. M.p. 145°.

Schmidt, Austin, *Ber.*, 1903, **36**, 3731.

Schmidt, Kämpf, *ibid.*, 3736.

**2-Nitrophenanthrene**



$C_{14}H_9O_2N$

MW, 223

Pale yellow cryst. from EtOH. M.p. 99°. Very sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ .  $CrO_3$  → 2-nitrophenanthraquinone.

Schmidt, Heinle, *Ber.*, 1911, **44**, 1494.

**3-Nitrophenanthrene.**

Dark yellow plates from AcOH. M.p. 170–1°. Mod. sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ , toluene. Spar. sol. cold EtOH, AcOH. Alc.  $(NH_4)_2S$  or  $SnCl_2$  in boiling AcOH → 3-aminophenanthrene.

Schmidt, *Ber.*, 1879, **12**, 1154.

See also Schmidt, *Ber.*, 1901, **34**, 3531.

**4-Nitrophenanthrene.**

Yellow needles from EtOH. M.p. 80–2°.  $CrO_3$  in AcOH → 4-nitrophenanthraquinone.  $SnCl_2$  in AcOH → 4-aminophenanthrene.

Schmidt, Heinle, *Ber.*, 1911, **44**, 1494.

**9-Nitrophenanthrene.**

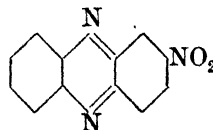
Pale yellow needles from EtOH. M.p. 116–17°. Sol.  $CHCl_3$ ,  $C_6H_6$ . Less sol. EtOH, MeOH,  $Et_2O$ . Spar. sol. ligroin. Sol. 49 parts AcOH at 17°. Sol. conc.  $H_2SO_4$  with blood-red col. → green on warming. Zn dust + alkali → 9-azophenanthrene.  $SnCl_2$  → 9-aminophenanthrene.

*Picrate*: pale yellow needles from EtOH. M.p. 78–9°.

Schmidt, Strobel, *Ber.*, 1903, **36**, 2511.

Schmidt, *Ber.*, 1900, **33**, 3257.

**2-Nitrophenazine**



$C_{12}H_7O_2N_3$

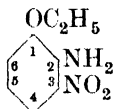
MW, 225



Yellow cryst. from EtOH. M.p. 214°. Sol. hot EtOH, hot AcOH. Insol. H<sub>2</sub>O. Sublimes. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with orange-yellow col.

Kehrmann, Havas, *Ber.*, 1913, **46**, 351.

**3-Nitro-*o*-phenetidine** (3-Nitro-2-amino-phenetole)



C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub> MW, 182

Yellow cryst. from H<sub>2</sub>O. M.p. 49°.

*N*-Me: C<sub>9</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 196. Dark red cryst. from EtOH. M.p. 59°.

*N*-Acetyl: cryst. from pet. ether. M.p. 64°.

Blanksma, *Chem. Zentr.*, 1908, **11**, 1826.

**4-Nitro-*o*-phenetidine.**

Yellow needles from EtOH.Aq. M.p. 99°. Sol. EtOH. Spar. sol. H<sub>2</sub>O.

*N*-Acetyl: yellow needles from EtOH. M.p. 199° (196°).

Reverdin, Düring, *Ber.*, 1899, **32**, 164.

See also previous reference.

**5-Nitro-*o*-phenetidine.**

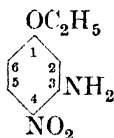
Yellow needles from EtOH.Aq. M.p. 91°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Spar. sol. ligroin.

*N*-Acetyl: yellow needles. M.p. 165°.

Reverdin, Düring, *Ber.*, 1899, **32**, 162.

Jacobson, Hönigsberger, *Ber.*, 1903, **36**, 4124.

**4-Nitro-*m*-phenetidine** (4-Nitro-3-amino-phenetole)



C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub> MW, 182

Yellow needles from EtOH.Aq. M.p. 105°. Sol. usual org. solvents.

*N*-Acetyl: needles from EtOH. M.p. 95°. Sol. usual org. solvents.

Reverdin, Lokietek, *Bull. soc. chem.*, 1916, **19**, 253.

Hodgson, Clay, *J. Chem. Soc.*, 1930, 963.

**5-Nitro-*m*-phenetidine.**

Yellow needles from EtOH. M.p. 115°. Sol. hot H<sub>2</sub>O, EtOH.

Blanksma, *Rec. trav. chim.*, 1905, **24**, 43.

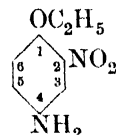
**6-Nitro-*m*-phenetidine.**

Needles from EtOH.Aq. M.p. 122-3°. Sol. usual org. solvents. Spar. sol. H<sub>2</sub>O. Insol. ligroin.

*N*-Acetyl: golden-yellow needles from H<sub>2</sub>O. M.p. 165° (147°). Sol. EtOH, AcOH. Spar. sol. H<sub>2</sub>O. Insol. ligroin.

Reverdin, Lokietek, *Bull. soc. chim.*, 1916, **19**, 253.

**2-Nitro-*p*-phenetidine** (2-Nitro-4-amino-phenetole)



C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub> MW, 182

Yellow needles from EtOH.Aq. M.p. 40°.

*N*-Acetyl: needles from EtOH. M.p. 123°. Very sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOH. Sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O, ligroin.

Reverdin, *Helv. Chim. Acta*, 1927, **10**, 3.

**3-Nitro-*p*-phenetidine.**

Red prisms from EtOH. M.p. 113° (109°). Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. cold EtOH.

*N*-Acetyl: yellow needles from H<sub>2</sub>O. M.p. 104°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

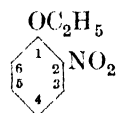
*N*-Benzenesulphonyl: yellow needles. M.p. 72°.

*N*-*p*-Toluenesulphonyl: yellow needles. M.p. 94°.

Kehrmann, Gauhe, *Ber.*, 1898, **31**, 2403.

Reverdin, *Ber.*, 1906, **39**, 3796.

***o*-Nitrophenetole**



C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N MW, 167

Yellow oil with green tinge. M.p. 2.1°. B.p. 267°, 149.3°/16 mm., 142.8°/12 mm., 134.2°/8 mm. D<sub>4</sub><sup>15</sup> 1.1903. n<sub>D</sub><sup>20</sup> 1.5425. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Francis, *Ber.*, 1906, **39**, 3803.

Erp, *Ber.*, 1923, **56**, 218.

Richardson, *J. Chem. Soc.*, 1926, 522.

***m*-Nitrophenetole.**

Yellow cryst. M.p. 34°. B.p. 284° (264° slight decomp.), 190°/100 mm., 169°/70 mm. Sn + HCl → *m*-phenetidine.

Whiston, *J. Soc. Chem. Ind.*, 1924, **43**, 369T.

Reverdin, *Ber.*, 1896, **29**, 2597.

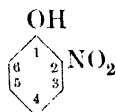
***p*-Nitrophenetole.**

Prisms from Et<sub>2</sub>O or EtOH.Aq. M.p. 60° (57-8°). B.p. 283°. D<sub>4</sub><sup>100</sup> 1.1176, D<sub>4</sub><sup>125</sup> 1.0937.

Very sol. Et<sub>2</sub>O. Sol. hot EtOH, hot pet. ether. Spar. sol. H<sub>2</sub>O, cold EtOH, cold pet. ether. Sn+dil. HCl or Fe+HCl → *p*-phenetidine. Sn+conc. HCl → 3-chloro-4-aminophenetole.

Gas, Light and Coke Co., E.P., 204,594, (Chem. Zentr., 1925, II, 611).  
Richardson, *J. Chem. Soc.*, 1926, 522.  
Willgerodt, *Ber.*, 1882, 15, 1003.  
de Bruyn, *Rec. trav. chim.*, 1904, 23, 36, 43.  
Erp, *Ber.*, 1923, 56, 218.

## o-Nitrophenol

C<sub>6</sub>H<sub>5</sub>O<sub>3</sub>N

MW, 139

Sulphur-yellow needles or prisms from EtOH or Et<sub>2</sub>O. M.p. 44.9°. B.p. 216°. D<sub>4</sub><sup>20</sup> 1.2942, D<sub>4</sub><sup>60</sup> 1.2712, D<sub>4</sub><sup>80</sup> 1.2482, D<sub>4</sub><sup>100</sup> 1.2323. Sol. EtOH, Et<sub>2</sub>O, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>, hot H<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O. Volatile in steam. Heat of comb. C<sub>p</sub> 688.2 Cal., C<sub>v</sub> 688.6 Cal.  $k=6.0 \times 10^{-8}$  at 0°,  $7.5 \times 10^{-8}$  (6.8 × 10<sup>-8</sup>) at 25°,  $8.3 \times 10^{-8}$  at 35°. Cold alk. ammonium persulphate → nitrohydroquinone.

*Me ether*: see *o*-Nitroanisole.

*Et ether*: see *o*-Nitrophenetole.

*Isobutyl ether*: C<sub>10</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 195. Yellow oil. B.p. 275–80°. D<sub>20</sub> 1.361.

*Allyl ether*: C<sub>9</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 179. Yellow oil. B.p. 155°/12 mm.

*β-Aminoethyl ether*: β-*o*-nitrophenoxyethylamine. C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 182. Red leaflets from H<sub>2</sub>O or plates from Et<sub>2</sub>O-C<sub>6</sub>H<sub>6</sub>. M.p. 72–3°. Very sol. EtOH, Et<sub>2</sub>O, boiling C<sub>6</sub>H<sub>6</sub>. Spar. sol. boiling H<sub>2</sub>O.

*Phenyl ether*: see 2-Nitrodiphenyl Ether.

*Benzyl ether*: C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 229. M.p. 130°.

*Acetyl*: *o*-nitrophenyl acetate. Needles or prisms from ligroin. M.p. 40–1°. B.p. 253° decomp. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin.

*Chloroacetyl*: needles. M.p. 63°.

*Bromoacetyl*: cryst. from EtOH. M.p. 55–56°.

*Isobutyryl*: *o*-nitrophenyl isobutyrate. Yellow oil. B.p. 163–4°/9 mm.

*Lauryl*: *o*-nitrophenyl laurate. Cryst. from EtOH. M.p. 35–6°.

*Palmityl*: *o*-nitrophenyl palmitate. Needles from EtOH. M.p. 51–2°.

*Stearyl*: *o*-nitrophenyl stearate. Cryst. from EtOH. M.p. 60–1°.

*Benzoyl*: *o*-nitrophenyl benzoate. Prisms from ligroin. M.p. 59° (55°).

*o-Nitrobenzoyl*: yellowish needles from EtOH. M.p. 125°.

*m-Nitrobenzoyl*: m.p. 129°.

*Anisoyl*: needles. M.p. 96°.

*p-Bromobenzenesulphonyl*: m.p. 97–8°.

*α-Naphthylurethane*: m.p. 112–13°.

Lukaschewitsch, Stenberg, *Chem. Zentr.*, 1935, II, 212.

Hart, *J. Am. Chem. Soc.*, 1910, 32, 1105.

Meisenheimer, Hesse, *Ber.*, 1919, 52, 1166.

Hantzsch, Gorke, *Ber.*, 1906, 39, 1080.

## m-Nitrophenol.

Cryst. from HCl.Aq. M.p. 97° (95.1°). B.p. 194°/70 mm. D<sub>4</sub><sup>100</sup> 1.2797, D<sub>4</sub><sup>25</sup> 1.2588, D<sub>4</sub><sup>100</sup> 1.2359. Heat of comb. C<sub>p</sub> 684.8 Cal.  $k=1.0 \times 10^{-8}$  at 25°. Very sol. EtOH, Et<sub>2</sub>O. Sol. hot H<sub>2</sub>O, hot CHCl<sub>3</sub>. Insol. pet. ether. Sol. dil. acids. Non-volatile in steam. Triboluminescent.

*Me ether*: see *m*-Nitroanisole.

*Et ether*: see *m*-Nitrophenetole.

*Phenyl ether*: see 3-Nitrodiphenyl Ether.

*Acetyl*: *m*-nitrophenyl acetate. Needles from pet. ether. M.p. 55–6°.

*Benzoyl*: *m*-nitrophenyl benzoate. Cryst. M.p. 95°.

*p-Bromobenzenesulphonyl*: m.p. 108–9°.

*p-Toluenesulphonyl*: prisms from EtOH. M.p. 112–13°.

*Phenylurethane*: needles from CHCl<sub>3</sub>. M.p. 129°.

*α-Naphthylurethane*: m.p. 167°.

Bamberger, *Ber.*, 1915, 48, 1355.

Manske, *Organic Syntheses*, Collective Vol. I, 396.

## p-Nitrophenol.

Known in two forms:

(α) Colourless prisms obtained by cryst. from toluene above 63°. Metastable at room temp. Stable to light.

(β) Yellow prisms by cryst. from toluene below 63°. Stable at room temp. Gradually turns red in light.

Ordinary *p*-nitrophenol is a mixture of both forms. M.p. 114°. Heat of comb. C<sub>p</sub> 689.1 Cal., C<sub>v</sub> 689.5 Cal.  $k=6.5 \times 10^{-8}$  at 25°. Very sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O. Sol. Na<sub>2</sub>CO<sub>3</sub>.Aq. Very spar. volatile in steam. Sublimes.

*Me ether*: see *p*-Nitroanisole.

*Et ether*: see *p*-Nitrophenetole.

*Propyl ether*: C<sub>9</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 181. Oil. B.p. 285–7° part. decomp. Sol. mos. org. solvents. Insol. H<sub>2</sub>O.

*Isobutyl ether*: C<sub>10</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 195.

Needles from Et<sub>2</sub>O. M.p. 39°. B.p. 293-5° decomp.

*Isomyl ether*: C<sub>11</sub>H<sub>15</sub>O<sub>3</sub>N. MW, 209. Oil. B.p. 309-10° decomp.

*Allyl ether*: C<sub>9</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 179. Needles. M.p. 36° (18.5°). B.p. 160°/12 mm.

*β-Aminoethyl ether*: β-*p*-nitrophenoxylethylamine. Yellow cryst. from H<sub>2</sub>O. M.p. 108-9°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Phenyl ether*: see 4-Nitrodiphenyl Ether.

*Benzyl ether*: C<sub>13</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 229. M.p. 187.4°.

*Acetyl*: *p*-nitrophenyl acetate. Leaflets from EtOH.Aq. M.p. 81-2°. Very sol. C<sub>6</sub>H<sub>6</sub>. Sol. hot H<sub>2</sub>O.

*Benzoyl*: *p*-nitrophenyl benzoate. Needles. M.p. 142.5°.

*o*-Nitrobenzoyl: needles. M.p. 111°.

*p*-Nitrobenzoyl: cryst. powder. M.p. 159°.

3 : 5-Dinitrobenzoyl: cryst. + 1AcOH. M.p. 188°.

*Anisoyl*: needles. M.p. 166°.

*p*-Bromobenzenesulphonyl: m.p. 112°.

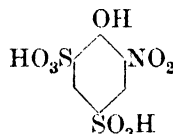
*α*-Naphthylurethane: m.p. 150-1°.

Aoyama, Nanai, *Chem. Zentr.*, 1935, II, 2662.

Robertson, *J. Chem. Soc.*, 1902, 81, 1477.

Pictet, Khotinsky, *Ber.*, 1907, 40, 1165.

**o-Nitrophenol-4 : 6-disulphonic Acid**



C<sub>6</sub>H<sub>5</sub>O<sub>9</sub>NS<sub>2</sub> MW, 299

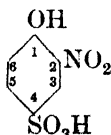
*K salt*: orange-yellow prisms + 1½H<sub>2</sub>O. FeCl<sub>3</sub> → violet-red col.

*Difluoride*: C<sub>6</sub>H<sub>3</sub>O<sub>7</sub>NF<sub>2</sub>S<sub>2</sub>. MW, 303. Cryst. M.p. 98.5-99.5°.

Chamot, Pratt, *J. Am. Chem. Soc.*, 1909, 31, 922; 1910, 32, 635.

Steinkopf et al., *J. prakt. Chem.*, 1927, 117, 1.

**o-Nitrophenol-4-sulphonic Acid**



C<sub>6</sub>H<sub>5</sub>O<sub>6</sub>NS MW, 219

Plates from AcOEt-C<sub>6</sub>H<sub>6</sub>, needles + 3H<sub>2</sub>O from hot H<sub>2</sub>O. M.p. 51.5°, anhyd. 141-2°. Sol. H<sub>2</sub>O. Very sol. EtOH, AcOEt, hot CHCl<sub>3</sub>.

*Me ether*: see *o*-Nitroanisole-4-sulphonic Acid.

*Phenyl ether*: 2-nitrodiphenyl ether 4-sulphonic acid. Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 89-90°.

*Fluoride*: C<sub>6</sub>H<sub>4</sub>O<sub>5</sub>NFS. MW, 221. Cryst. M.p. 66-7°.

Gnehm, Knecht, *J. prakt. Chem.*, 1906, 73, 521.

Kolbe, Gauhe, *Ann.*, 1868, 147, 71.

Maqueyrol, Lorette, *Bull. soc. chim.*, 1919, 25, 371.

Steinkopf et al., *J. prakt. Chem.*, 1927, 117, 1.

**m-Nitrophenol-4-sulphonic Acid.**

*K salt*: yellow leaflets from hot H<sub>2</sub>O.

Nietzki, Helbach, *Ber.*, 1896, 29, 2450.

**p-Nitrophenol-2-sulphonic Acid.**

Prisms or needles or plates + 3H<sub>2</sub>O. De-comp. at 110°. FeCl<sub>3</sub> → deep reddish-brown col. Easily decomp.

*K salt*: orange-red needles + H<sub>2</sub>O. Very sol. H<sub>2</sub>O.

*Ca salt*: prisms + 3H<sub>2</sub>O.

*Ba salt*: needles or prisms + H<sub>2</sub>O.

*Pb salt*: needles + 1½H<sub>2</sub>O. Insol. H<sub>2</sub>O.

Ullmann, Dahmen, *Ber.*, 1908, 41, 3755.

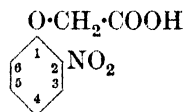
Post, Stuckenberg, *Ann.*, 1880, 205, 41.

**p-Nitrophenol-3-sulphonic Acid.**

Cryst. Fe + AcOH → *p*-aminophenol-3-sulphonic acid.

Kalle, D.R.P., 153,123, (*Chem. Zentr.*, 1904, II, 574).

**o-Nitrophenoxycetic Acid (Glycollic acid 2-nitrophenyl ether)**



C<sub>8</sub>H<sub>7</sub>O<sub>5</sub>N MW, 197

Prisms from H<sub>2</sub>O. M.p. 156.5°. Sol. EtOH. *k* = 1.58 × 10<sup>-3</sup> at 25°. Non-volatile in steam.

*Me ester*: C<sub>9</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 211. Needles. M.p. 58°.

*Et ester*: C<sub>10</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 225. Needles or leaflets from EtOH.Aq.. M.p. 46-7°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*Chloride*: C<sub>8</sub>H<sub>6</sub>O<sub>4</sub>NCl. MW, 215.5. Yellowish needles from ligroin, prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 43-44.5°. Sol. most org. solvents.

*Amide*: C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub>. MW, 196. Needles from H<sub>2</sub>O. M.p. 194.5-199.5° (188°). Spar. sol. hot H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO.

*Anilide*:  $C_{14}H_{12}O_4N_2$ . MW, 272. Yellow leaflets. M.p.  $118.5^\circ$ .

Minton, Stephen, *J. Chem. Soc.*, 1922, **121**, 1591.

Jacobs, Heidelberger, *J. Am. Chem. Soc.*, 1917, **39**, 2421.

**m-Nitrophenoxyacetic Acid** (*Glycollic acid 3-nitrophenyl ether*).

Yellowish needles from  $H_2O$ . M.p.  $154.5^\circ$  ( $151^\circ$ ). Sol. AcOH. Mod. sol.  $CHCl_3$ ,  $C_6H_6$ , toluene.

*Me ester*: needles. M.p.  $66.5^\circ$ .

*Et ester*: yellow liq. B.p.  $208-12^\circ/30$  mm.,  $187^\circ/14$  mm.

*Chloride*: prisms from  $C_6H_6$ . M.p.  $49-51^\circ$ .

*Amide*: yellow prisms. M.p.  $178.5^\circ$ .

*Anilide*: needles. M.p.  $125^\circ$ .

Minton, Stephen, *J. Chem. Soc.*, 1922, **121**, 1591.

Hewitt, Johnson, Pope, *J. Chem. Soc.*, 1913, **103**, 1631.

Jacobs, Heidelberger, *J. Am. Chem. Soc.*, 1917, **39**, 2191.

Meyer, Duczmal, *Ber.*, 1913, **46**, 3377.

**p-Nitrophenoxyacetic Acid** (*Glycollic acid 4-nitrophenyl ether*).

Leaflets from MeOH or EtOH. M.p.  $184^\circ$ . Sol. hot EtOH. Spar. sol.  $H_2O$ .  $k = 1.53 \times 10^{-3}$  at  $25^\circ$ .

*Me ester*: needles. M.p.  $100-1^\circ$  ( $99^\circ$ ).

*Et ester*: pale yellow leaflets from EtOH. M.p.  $75-6^\circ$ . B.p.  $203-6^\circ/15$  mm. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Propyl ester*: m.p.  $75-6^\circ$ .

*Amide*: prisms or needles. M.p.  $156-8^\circ$  ( $154-6^\circ$ ). Sol. hot EtOH. Spar. sol.  $H_2O$ .

*Methylamide*: needles from  $H_2O$ . M.p.  $165.5^\circ$ . Mod. sol. hot  $H_2O$ . Less sol. EtOH, toluene.

*Chloride*: plates or prisms from  $C_6H_6$ . M.p.  $86-7^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ .

*Anilide*: yellow leaflets. M.p.  $170^\circ$ .

*p-Phenetidide*: needles. M.p.  $156-7^\circ$ . Spar. sol. EtOH.

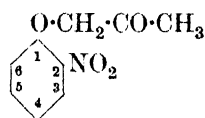
Jacobs, Heidelberger, *J. Am. Chem. Soc.*, 1917, **39**, 2424.

Minton, Stephen, *J. Chem. Soc.*, 1922, **121**, 1591.

Fuchs, D.R.P., 96,492, (*Chem. Zentr.*, 1898, I, 1252).

A.G.F.A., D.R.P., 108,342, (*Chem. Zentr.*, 1900, I, 1177).

**o-Nitrophenoxyacetone**



$C_9H_9O_4N$

MW, 195

Needles from  $H_2O$ . M.p.  $69^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ . Mod. sol. hot  $H_2O$ , EtOH. Non-volatile in steam. Gives cryst. bisulphite comp. *Oxime*: needles. M.p.  $102^\circ$ . Sol. warm EtOH. Spar. sol.  $H_2O$ .

*Semicarbazone*: cryst. powder. M.p.  $178^\circ$ .

*Phenylhydrazone*: yellow plates. M.p.  $101^\circ$ .

Stoermer, Brockerhof, *Ber.*, 1897, **30**, 1634.

M.L.B., D.R.P., 97,242, (*Chem. Zentr.*, 1898, II, 525).

**p-Nitrophenoxyacetone**.

Yellowish leaflets from  $H_2O$  or EtOH. M.p.  $81^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ , EtOH. Gives cryst. bisulphite comp.

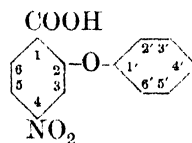
*Oxime*: cryst. M.p.  $119^\circ$ . Mod. sol. hot  $H_2O$ , EtOH.

*Semicarbazone*: cryst. powder from EtOH. Aq. M.p.  $216^\circ$  decomp. Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ .

*Phenylhydrazone*: yellow needles from EtOH. M.p.  $140^\circ$ .

See first reference above.

**4-Nitro-o-phenoxybenzoic Acid** (*5-Nitro-diphenyl ether 2-carboxylic acid*)



$C_{13}H_9O_5N$

MW, 259

Plates from  $C_6H_6$ . M.p.  $156^\circ$ . Sol. EtOH, AcOH, hot  $C_6H_6$ . Insol.  $H_2O$ , ligroin. Conc.  $H_2SO_4$  at  $100^\circ \rightarrow$  2-nitroxanthone.

Ullmann, Wagner, *Ann.*, 1907, **355**, 361.

**5-Nitro-o-phenoxybenzoic Acid** (*4-Nitro-diphenyl ether 2-carboxylic acid*).

Cryst. from  $H_2O$ . M.p.  $171-2^\circ$  ( $168^\circ$ ). Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Conc.  $H_2SO_4 \rightarrow$  3-nitroxanthone.

Haeussermann, Bauer, *Ber.*, 1897, **30**, 740.

Purgotti, *Gazz. chim. ital.*, 1914, **44**, 643.

Brewster, Strain, *J. Am. Chem. Soc.*, 1934, **56**, 118.

**2'-Nitro-*o*-phenoxybenzoic Acid** (2'-*Nitro-diphenyl ether 2-carboxylic acid*).

Cryst. from  $C_6H_6$ . M.p. 153°.

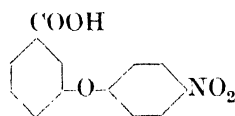
Brewster, Strain, *J. Am. Chem. Soc.*, 1934, **56**, 118.

**4'-Nitro-*o*-phenoxybenzoic Acid** (4'-*Nitro-diphenyl ether 2-carboxylic acid*).

Cryst. from  $C_6H_6$ . M.p. 157°. Conc.  $H_2SO_4$  → 3-nitroxanthone.

See previous reference.

**4'-Nitro-*m*-phenoxybenzoic Acid** (4'-*Nitrodiphenyl ether 3-carboxylic acid*)



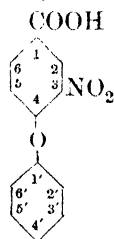
$C_{13}H_9O_5N$

MW, 259

Prisms from dil. AcOH. M.p. 183°.

Scarborough, Sweeten, *J. Chem. Soc.*, 1934, 54.

**3-Nitro-*p*-phenoxybenzoic Acid** (2-*Nitro-diphenyl ether 4-carboxylic acid*)



$C_{13}H_9O_5N$

MW, 259

M.p. 174-5°. Sol. EtOH,  $Et_2O$ . Mod. sol.  $C_6H_6$ . Spar. sol. hot  $H_2O$ .

*Et ester*:  $C_{15}H_{13}O_5N$ . MW, 287. Yellow cryst. from EtOH. M.p. 93-4°.

*Nitrile*:  $C_{13}H_8O_3N_2$ . MW, 240. Yellow prisms from MeOH. M.p. 79°.

Haeussermann, Bauer, *Ber.*, 1897, **30**, 739. Borscæ, *Ber.*, 1923, **56**, 1490.

**2'-Nitro-*p*-phenoxybenzoic Acid** (2'-*Nitro-diphenyl ether 4-carboxylic acid*).

Needles from  $H_2O$  or xylene. M.p. 184° (182-3°). Sol. EtOH, AcOH. Spar. sol.  $Et_2O$ . Insol. pet ether.

*Me ester*:  $C_{14}H_{11}O_5N$ . MW, 273. Yellow prisms from EtOH. M.p. 88°.

Cool, Hillyer, *Am. Chem. J.*, 1900, **24**, 57.

Mayer, Krieger, *Ber.*, 1922, **55**, 1663, (Apotnote).

**4'-Nitro-*p*-phenoxybenzoic Acid** (4'-*Nitro-diphenyl ether 4-carboxylic acid*).

Prisms from boiling EtOH or plates from AcOH.

M.p. 245° (236-7°). Spar. sol. hot EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $H_2O$ .

*Me ester*: needles from MeOH. M.p. 108-9°.

*Et ester*: needles from MeOH. M.p. 78° (74-5°).

*Chloride*:  $C_{13}H_8O_4NCl$ . MW, 277.5. Needles from pet. ether. M.p. 79-80°.

*Amide*:  $C_{13}H_{10}O_4N_2$ . MW, 258. M.p. 167-8°.

Haeussermann, Bauer, *Ber.*, 1896, **29**, 2084.

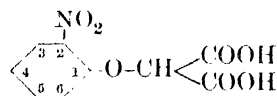
Suter, Oberg, *J. Am. Chem. Soc.*, 1931, **53**, 1567.

Scarborough, Sweeten, *J. Chem. Soc.*, 1934, 55.

**Nitrophenoxyethylamine.**

*See under Nitrophenol.*

***o*-Nitrophenoxy malonic Acid**



$C_9H_7O_7N$

MW, 241

*Di-Me ester*:  $C_{11}H_{11}O_7N$ . MW, 269. Needles from MeOH. M.p. 123°. Sol. most org. solvents. Spar. sol. cold MeOH.

*Di-Et ester*:  $C_{13}H_{15}O_7N$ . MW, 297. Needles from  $C_6H_6$ . M.p. 116-18°.

Bischoff, *Ber.*, 1907, **40**, 3139.

***m*-Nitrophenoxy malonic Acid.**

*Di-Me ester*: yellowish leaflets from MeOH. M.p. 100°.

*Di-Et ester*: leaflets. M.p. 78°. Sol.  $C_6H_6$ .

See previous reference.

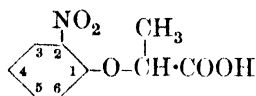
***p*-Nitrophenoxy malonic Acid.**

Needles from  $Et_2O$ . Sinters at 160°. Sol.  $Et_2O$ ,  $Me_2CO$ , AcOH. Mod. sol.  $H_2O$ , EtOH. Spar. sol.  $C_6H_6$ , ligroin,  $CHCl_3$ . Decomp. at 168-70° → *p*-nitrophenoxyacetic acid. Impure acid decomp. slowly on standing.

*Di-Me ester*: pale yellow prisms or needles. M.p. 101°. B.p. 221-2°/15 mm., slight decomp. Sol.  $CHCl_3$ , AcOH, AcOEt. Mod. sol. EtOH,  $C_6H_6$ . Spar. sol.  $Et_2O$ , ligroin.

*Di-Et ester*: needles from MeOH. M.p. 86°. B.p. 241-2°/15 mm., slight decomp. Sol.  $CHCl_3$ , AcOH. Mod. sol. EtOH,  $C_6H_6$ . Spar. sol.  $Et_2O$ , ligroin.

See previous reference.

**1-o-Nitrophenoxypropionic Acid** (*Lactic acid 2-nitrophenyl ether*) $C_9H_9O_5N$ 

MW, 211

*d.*

Cryst. from EtOH.Aq. M.p. 111–12°.  $[\alpha]_D^{21} + 166.25^\circ$  in EtOH.

*l.*

Cryst. from EtOH.Aq. M.p. 111–12°.  $[\alpha]_D^{21} - 166.00^\circ$ .

*dl.*

Pale yellow needles from EtOH.Aq. M.p. 157–9°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. cold H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

*Et ester*: C<sub>11</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 225. Needles from EtOH. M.p. 48°. Sol. most org. solvents. Spar. sol. H<sub>2</sub>O.

*o-Nitrophenyl ester*: prisms from EtOH. M.p. 137°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, ligroin.

Bischoff, *Ber.*, 1900, **33**, 930, 1593; 1906, **39**, 3858.

Fourneau, Sandulesco, *Bull. soc. chim.*, 1922, **31**, 988.

**1-m-Nitrophenoxypropionic Acid** (*Lactic acid 3-nitrophenyl ether*).*d.*

Yellow needles from H<sub>2</sub>O. M.p. 101–2°.  $[\alpha]_D^{21} + 51.8^\circ$ .

*l.*

Yellow needles from H<sub>2</sub>O. M.p. 101–2°.  $[\alpha]_D^{21} - 51.87^\circ$ .

*Strychnine salt*:  $[\alpha]_D^{21} - 25^\circ$ .

*dl.*

Yellow needles. M.p. 109–10°. Sol. H<sub>2</sub>O, ligroin. Decomp. slowly in air.

*Me ester*: b.p. 173–5°/20 mm.

*Et ester*: oil. B.p. 295–6°/769 mm. slight decomp., 187°/7 mm.

*m-Nitrophenyl ester*: cryst. from MeOH. M.p. 109–10°. Spar. sol. MeOH, EtOH. Insol. H<sub>2</sub>O, ligroin.

Bischoff, *Ber.*, 1900, **33**, 1598; 1906, **39**, 3859.

Fourneau, Sandulesco, *Bull. soc. chim.*, 1923, **33**, 459.

**1-p-Nitrophenoxypropionic Acid** (*Lactic acid 4-nitrophenyl ether*).*d.*

Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 88–90°.  $[\alpha]_D^{21} + 53.7^\circ$ . Very sol. EtOH, Me<sub>2</sub>CO.

*Quinidine salt*:  $[\alpha]_D^{21} + 172.5^\circ$ .

*l.*

Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 89–90°.  $[\alpha]_D^{21} - 53.7^\circ$ .

*Yohimbine salt*:  $[\alpha]_D^{21} + 20.6^\circ$ .

*dl.*

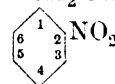
Needles from EtOH. M.p. 142.5–143°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. H<sub>2</sub>O, CS<sub>2</sub>, CHCl<sub>3</sub>, ligroin.

*Et ester*: needles from EtOH. M.p. 59–61.5°. B.p. 195.5°/4 mm.

*p-Nitrophenyl ester*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 137°. Sol. usual org. solvents.

See second reference above and also

Bischoff, *Ber.*, 1900, **33**, 930, 1600; 1906, **39**, 3861.

**o-Nitrophenylacetaldehyde**CH<sub>2</sub>·CHO $C_8H_7O_3N$ 

MW, 165

Yellowish plates. M.p. 22–3°. B.p. 133–5°/5 mm. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold H<sub>2</sub>O, CCl<sub>4</sub>. Insol. ligroin. Fe powder + NaHSO<sub>3</sub> → indole. Alkalis → red col.

*Oxime*: needles from H<sub>2</sub>O. M.p. 110°. Very sol. EtOH, Et<sub>2</sub>O. Sol. C<sub>6</sub>H<sub>6</sub>.

Weermann, *Ann.*, 1913, **401**, 13.

**m-Nitrophenylacetaldehyde.**

Needles from H<sub>2</sub>O or damp ether. M.p. 78–9°. Sol. EtOH. Mod. sol. Et<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>. Alkalis → yellow col. Volatile in steam.

*Oxime*: leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 105–6°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

See previous reference.

**p-Nitrophenylacetaldehyde.**

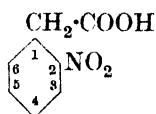
Needles. M.p. 85–6°. Very sol. EtOH, Et<sub>2</sub>O. Sol. hot H<sub>2</sub>O. Alkalis → deep red col.

*Oxime*: needles from Et<sub>2</sub>O. M.p. 155°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O, ligroin.

Lipp, *Ber.*, 1886, **19**, 2647.

See also previous reference.

## o-Nitrophenylacetic Acid



$\text{C}_8\text{H}_7\text{O}_4\text{N}$  MW, 181

Needles from EtOH. M.p.  $141^\circ$  ( $137-8^\circ$ ).  
 $\text{KMnO}_4 \longrightarrow$  o-nitrobenzoic acid.

*Me ester*:  $\text{C}_9\text{H}_9\text{O}_4\text{N}$ . MW, 195. Liq. B.p.  $264^\circ$ .

*Et ester*:  $\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$ . MW, 209. Cryst. from 95% EtOH. M.p.  $69^\circ$ . Sol. usual org. solvents.

*Amide*:  $\text{C}_8\text{H}_8\text{O}_3\text{N}_2$ . MW, 180. Plates or needles from EtOH or  $\text{C}_6\text{H}_6$ . M.p.  $160-1^\circ$ .

*Nitrile*: see o-Nitrobenzyl cyanide.

Reissert, *Ber.*, 1897, **30**, 1041; 1908, **41**, 3814, 3925.

Reissert, Scherk, *Ber.*, 1898, **31**, 395.

Pschorr, Hoppe, *Ber.*, 1910, **43**, 2547.

## m-Nitrophenylacetic Acid.

Needles from  $\text{H}_2\text{O}$ . M.p.  $120^\circ$  ( $117^\circ$ ).

*Amide*: cryst. from  $\text{H}_2\text{O}$ . M.p.  $109-10^\circ$ . Sol. EtOH,  $\text{C}_6\text{H}_6$ .

*Nitrile*: see m-Nitrobenzyl cyanide.

Salkowski, *Ber.*, 1884, **17**, 506.

Heller, *Ann.*, 1908, **358**, 357.

Purgotti, *Gazz. chim. ital.*, 1890, **20**, 596.

## p-Nitrophenylacetic Acid.

Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $151-2^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ .  $k = 1.04 \times 10^{-4}$  at  $25^\circ$ .  $\text{Sn} + \text{HCl} \longrightarrow$  p-amino-phenylacetic acid.  $\text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{SO}_4 \longrightarrow$  p-nitrobenzoic acid.

*Me ester*: needles from ligroin. M.p.  $54^\circ$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Alc. KOH  $\longrightarrow$  violet col.

*Et ester*: leaflets from ligroin. M.p.  $69^\circ$  ( $65^\circ$ ). B.p.  $196-7^\circ/20$  mm. Sol. EtOH,  $\text{Et}_2\text{O}$ . Less sol. ligroin.

*Benzyl ester*:  $\text{C}_{15}\text{H}_{13}\text{O}_4\text{N}$ . MW, 271. Needles from pet. ether. M.p.  $92^\circ$ .

*1-Naphthyl ester*:  $\text{C}_{18}\text{H}_{13}\text{O}_4\text{N}$ . MW, 307. Yellow needles from AcOH, m.p.  $152^\circ$ ; yellow plates from EtOH, m.p.  $146^\circ$ .

*Chloride*:  $\text{C}_8\text{H}_8\text{O}_3\text{NCl}$ . MW, 199.5. Cryst. from  $\text{CS}_2$ -ligroin, pale yellow plates from pet. ether. M.p.  $46-7^\circ$ . B.p.  $135-8^\circ/0.1$  mm. Sol.  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin.

*Amide*: prisms. M.p.  $197-8^\circ$  ( $191^\circ$ ). Spar. sol. EtOH. Very spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*Methylamide*:  $\text{C}_9\text{H}_{10}\text{O}_3\text{N}_2$ . MW, 194. Cryst. from  $\text{Me}_2\text{CO}$  or  $\text{CHCl}_3$ . M.p.  $159^\circ$ . Very sol. most org. solvents. Spar. sol.  $\text{H}_2\text{O}$ , pet. ether.

*Dimethylamide*:  $\text{C}_{10}\text{H}_{12}\text{O}_3\text{N}_2$ . MW, 208. Cryst. from  $\text{Et}_2\text{O}$ . M.p.  $90-1^\circ$ .

*Ethylamide*:  $\text{C}_{10}\text{H}_{12}\text{O}_3\text{N}_2$ . MW, 208. Cryst. M.p.  $155^\circ$ .

*Anilide*:  $\text{C}_{14}\text{H}_{12}\text{O}_3\text{N}_2$ . MW, 256. Cryst. M.p.  $198^\circ$ .

*Nitrile*: see p-Nitrobenzyl cyanide.

*Hydrazide*: yellowish needles from EtOH. M.p.  $167^\circ$ . Mod. sol. hot EtOH. Spar. sol. hot  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . *B, HCl*: yellowish needles from EtOH. M.p.  $251^\circ$  decomp. Very sol.  $\text{H}_2\text{O}$ .

*Azide*: cryst. M.p.  $45^\circ$  decomp. Explodes on rapid heating.

Taverne, *Rec. trav. chim.*, 1897, **16**, 35, 254.

Purgotti, *Gazz. chim. ital.*, 1890, **20**, 595.

Borsche, *Ber.*, 1909, **42**, 3596.

Maxwell, *Ber.*, 1879, **12**, 1765.

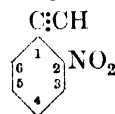
Pschorr, Wolfes, Buckow, *Ber.*, 1900, **33**, 170.

Curtius, *J. prakt. Chem.*, 1914, **89**, 522.

Wedekind, *Ann.*, 1911, **378**, 289.

Robertson, *Organic Syntheses*, Collective Vol. I, 389, 398.

## o-Nitrophenylacetylene



$\text{C}_8\text{H}_5\text{O}_2\text{N}$  MW, 147

Needles from EtOH.Aq. M.p.  $81-2^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH. Unpleasant odour. Volatile in steam.  $\text{NH}_3\cdot\text{AgNO}_3 \longrightarrow$  yellowish-white ppt.  $\text{NH}_3\cdot\text{CuCl}_2 \longrightarrow$  red ppt.

Baeyer, *Ber.*, 1880, **13**, 2259.

Kippenberg, *Ber.*, 1897, **30**, 1130.

## m-Nitrophenylacetylene.

Yellow cryst. M.p.  $27^\circ$ . B.p.  $120^\circ/11$  mm. Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol. AcOH. Volatile in steam. Explodes on dist. under ord. press.  $\text{NH}_3\cdot\text{AgNO}_3 \longrightarrow$  greenish-yellow ppt.  $\text{NH}_3\cdot\text{CuCl}_2 \longrightarrow$  reddish-brown ppt.

Reich, Koehler, *Ber.*, 1913, **46**, 3737.

Wollring, *Ber.*, 1914, **47**, 111.

## p-Nitrophenylacetylene.

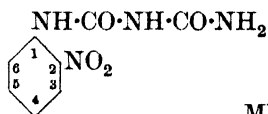
Needles from hot  $\text{H}_2\text{O}$ . M.p.  $152^\circ$  ( $149^\circ$ ). Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , AcOH,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Mod. sol. hot  $\text{H}_2\text{O}$ . Spar. sol. ligroin. Turns brown in air.  $\text{NH}_4\text{AgNO}_3 \longrightarrow$  yellow ppt.  $\text{NH}_3\cdot\text{CuCl}_2 \longrightarrow$  deep red ppt.

Drewsen, *Ann.*, 1882, **212**, 158.

Müller, *ibid.*, 136.

Wieland, *Ann.*, 1903, **328** 233.

**$\omega$ -o-Nitrophenylbiuret** (*Allophanic o-nitroanilide*)


 $\text{C}_8\text{H}_8\text{O}_4\text{N}_4$ 

MW, 224

Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $181^\circ$ .

Pickard, Allen, Bowdler, Carter, *J. Chem. Soc.*, 1902, **81**, 1568.

**$\omega$ -m-Nitrophenylbiuret** (*Allophanic m-nitroanilide*).

Yellow cryst. from EtOH.Aq. M.p.  $178^\circ$ . Spar. sol. NaOH.

See previous reference.

**$\omega$ -p-Nitrophenylbiuret** (*Allophanic p-nitroanilide*).

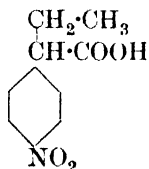
Golden-yellow needles from  $\text{H}_2\text{O}$ . M.p.  $206^\circ$ . Sol. EtOH.

See previous reference.

**Nitrophenylbutane.**

See Nitrobutylbenzene.

**1-p-Nitrophenylbutyric Acid**


 $\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$ 

MW, 209

*d.*

Cryst. from  $\text{H}_2\text{O}$ . M.p.  $120-2^\circ$ .  $[\alpha]_D^{25} + 17.7^\circ$  in AcOEt.

*Quinine salt*: cryst. from MeOH. M.p.  $183-5^\circ$ .  $[\alpha]_D^{25} - 42.2^\circ$  in Py.

*l.*

Cryst. from MeOH. M.p.  $120-2^\circ$ .  $[\alpha]_D^{25} - 17.8^\circ$  in AcOEt.

*Quinine salt*: cryst. from MeOH. M.p.  $180-2^\circ$ .  $[\alpha]_D^{25} - 53.8^\circ$  in Py.

*dl.*

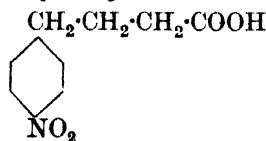
Cryst. from AcOH.Aq. M.p.  $118-20^\circ$ .

Chu, Marvel, *J. Am. Chem. Soc.*, 1933, **55**, 2841.

**2-p-Nitrophenylbutyric Acid.**

See p-Nitro- $\beta$ -methylhydrocinnamic Acid.

**3-p-Nitrophenylbutyric Acid**

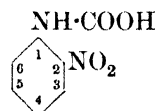

 $\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$ 

MW, 209

Prisms from  $\text{C}_6\text{H}_6$ . M.p.  $92-3^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ , pet. ether.

Scheer, *J. Am. Chem. Soc.*, 1934, **56**, 744.

**o-Nitrophenylcarbamic Acid** (*o-Nitrocarbanilic acid*)


 $\text{C}_7\text{H}_6\text{O}_4\text{N}_2$ 

MW, 182

*Me ester*:  $\text{C}_8\text{H}_8\text{O}_4\text{N}_2$ . MW, 196. Greenish-yellow cryst. from pet. ether. M.p.  $53^\circ$ .

*Et ester*: see o-Nitrophenylurethane.

*Isopropyl ester*:  $\text{C}_{10}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 224. Cryst. from pet. ether. M.p.  $12^\circ$ .

*Isobutyl ester*:  $\text{C}_{11}\text{H}_{14}\text{O}_4\text{N}_2$ . MW, 238. Yellow cryst. M.p.  $13^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ .

*Amyl ester*:  $\text{C}_{12}\text{H}_{16}\text{O}_4\text{N}_2$ . MW, 252. Cryst. from pet. ether. M.p.  $-5^\circ$ .

*Chloride*:  $\text{C}_7\text{H}_5\text{O}_3\text{N}_2\text{Cl}$ . MW, 200.5. Needles from pet. ether. M.p.  $47^\circ$ . Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether.

*Amide*: see o-Nitrophenylurea.

*Anilide*: see 2-Nitrocarbanilide.

*Nitrile*: see o-Nitrophenylcyanamide.

Folin, *Am. Chem. J.*, 1897, **19**, 326.

Swartz, *ibid.*, 303.

**m-Nitrophenylcarbamic Acid** (*m-Nitrocarbanilic acid*).

*Me ester*: yellow cryst. M.p.  $147-9^\circ$ .

*Et ester*: see m-Nitrophenylurethane.

*Chloride*: cryst. M.p.  $102^\circ$  decomp.

*Amide*: see m-Nitrophenylurea.

*Anilide*: see 3-Nitrocarbanilide.

*Nitrile*: see o-Nitrophenylcyanamide.

Folin, *Am. Chem. J.*, 1897, **19**, 325.

**p-Nitrophenylcarbamic Acid** (*p-Nitrocarbanilic acid*).

*Me ester*: m.p.  $179.5^\circ$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol. ligroin. N-Me: needles. M.p.  $110-11^\circ$ .

*Et ester*: see p-Nitrophenylurethane.

*Propyl ester*: m.p.  $115^\circ$ .

*Isopropyl ester*: m.p.  $116^\circ$  ( $78^\circ$ ).

*Butyl ester*: m.p.  $95.5^\circ$ .

*Isobutyl ester*: m.p.  $80^\circ$  ( $62^\circ$ ).

*Chloride*: cryst. Decomp. on heating.

*Amide*: see p-Nitrophenylurea.

*Anilide*: see 4-Nitrocarbanilide.

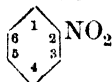
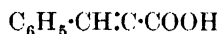
*Nitrile*: see p-Nitrophenylcyanamide.

Romburgh, *Rec. trav. chim.*, 1929, **48**, 922.

Shriner, Cox, *J. Am. Chem. Soc.*, 1931, **53**, 1604.



**$\alpha$ -o-Nitrophenylcinnamic Acid** (2-Nitro-stilbene- $\alpha$ -carboxylic acid)



$\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}$

MW, 269

Pale yellow needles from EtOH. M.p. 193°.

Borsche, *Ber.*, 1909, **42**, 3601.

**$\alpha$ -p-Nitrophenylcinnamic Acid** (4-Nitro-stilbene- $\alpha$ -carboxylic acid).

Needles from AcOH. M.p. 224-5°. Sol.  $\text{Et}_2\text{O}$ , hot EtOH, AcOH,  $\text{C}_6\text{H}_6$ .

*Me ester*:  $\text{C}_{16}\text{H}_{13}\text{O}_4\text{N}$ . MW, 283. Needles from EtOH. M.p. 104°.

*Et ester*:  $\text{C}_{17}\text{H}_{15}\text{O}_4\text{N}$ . MW, 297. Needles from EtOH. M.p. 86°.

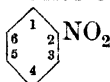
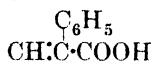
*Nitrile*:  $\text{C}_{15}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 250. Yellow needles from EtOH. M.p. 175-6°. Sol.  $\text{CHCl}_3$ , AcOH,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

Borsche, *Ber.*, 1909, **42**, 3597.

v. Walther, Wetzlich, *J. prakt. Chem.*, 1900, **61**, 181.

Remse, *Ber.*, 1890, **23**, 3134.

### 2-Nitro- $\alpha$ -phenylcinnamic Acid



$\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}$

MW, 269

*Cis*:

Short yellow prisms from EtOH or  $\text{C}_6\text{H}_6$ . M.p. 146-7°. Stable to most reagents.

*Me ester*:  $\text{C}_{16}\text{H}_{13}\text{O}_4\text{N}$ . MW, 283. Yellow needles from EtOH. M.p. 95-6°.

*Amide*:  $\text{C}_{15}\text{H}_{12}\text{O}_3\text{N}_2$ . MW, 268. Yellow cryst. from  $\text{CHCl}_3$ -pet. ether. M.p. 166-7°.

*Anilide*: yellow cryst. powder from EtOH. M.p. 148-9°.

*p-Toluidide*: m.p. 181-2°.

*Trans*:

Yellow cryst. from EtOH. M.p. 195-6°. Mod. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{H}_2\text{O}$ , EtOH. Sol. 14 parts hot toluene.  $\text{FeSO}_4 + \text{NH}_3$ . Aq.  $\rightarrow$  2-amino- $\alpha$ -phenylcinnamic acid.

*Me ester*: yellow prisms from EtOH. M.p. 75-6°.

*Et ester*:  $\text{C}_{17}\text{H}_{15}\text{O}_4\text{N}$ . MW, 297. Prisms from pet. ether. M.p. 59°. Mod. sol. cold EtOH.

*o-Tolyl ester*:  $\text{C}_{22}\text{H}_{17}\text{O}_4\text{N}$ . MW, 359. Yellow prisms from EtOH. M.p. 97-8°. Mod. sol. EtOH.

*Chloride*:  $\text{C}_{15}\text{H}_{10}\text{O}_3\text{NCl}$ . MW, 287.5. M.p. 100°.

*Amide*: pale yellow needles from toluene. M.p. 173-4°.

*Nitrile*:  $\text{C}_{15}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 250. Yellow needles. M.p. 127-8°.

*Anhydride*: yellow prisms from EtOH. M.p. 126°. Spar. sol. hot EtOH.

*Anilide*: yellow needles from EtOH. M.p. 136°.

Pschorr, *Ber.*, 1896, **29**, 497.

Bakunin, Parlatti, *Gazz. chim. ital.*, 1906, **36**, ii, 274.

Bakunin, *Gazz. chim. ital.*, 1895, **25**, i, 139, 182; 1897, **27**, ii, 36, 48.

Stoermer, Prigge, *Ann.*, 1915, **409**, 20.

### 3-Nitro- $\alpha$ -phenylcinnamic Acid.

*Cis*:

Needles from EtOH. M.p. 195-6°. Direct sunlight  $\rightarrow$  *trans*-form.

*Me ester*: leaflets from EtOH. M.p. 115-16°. Sol. hot EtOH.

*o-Tolyl ester*: needles from EtOH. M.p. 83-4°.

*Anhydride*: needles from EtOH. M.p. 129°.

*Anilide*: m.p. 161-2°.

*p-Toluidide*: m.p. 143-4°.

*Trans*:

Yellow prisms from  $\text{Et}_2\text{O}$ . M.p. 181-2°. Sol. boiling EtOH. Mod. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ , pet. ether. In sunlight partially converted to *cis*-form.

*Me ester*: yellowish prisms from EtOH or  $\text{Et}_2\text{O}$ . M.p. 78-9°. Sol. pet. ether.

*o-Tolyl ester*: cryst. from EtOH. M.p. 118-20°.

*Nitrile*: yellow cryst. M.p. 133-4°.

*Anhydride*: yellow needles from  $\text{Me}_2\text{CO}$ . M.p. 151°. Mod. sol.  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. pet. ether.

Bakunin, *Gazz. chim. ital.*, 1895, **25**, i, 145, 175; 1897, **27**, ii, 36; 1902, **32**, i, 180.

See also Bakunin, *Gazz. chim. ital.*, 1900, **30**, ii, 353; 1901, **31**, ii, 83.

### 4-Nitro- $\alpha$ -phenylcinnamic Acid.

*Cis*:

Yellowish-green prisms +  $\text{H}_2\text{O}$  from EtOH. Aq., yellow leaflets +  $\frac{1}{4}\text{C}_6\text{H}_6$  from  $\text{C}_6\text{H}_6$ . M.p. 144°. Very sol. EtOH. Mod. sol. other solvents. Sunlight  $\rightarrow$  *trans*-form.

*Me ester*: needles from EtOH. M.p. 147–148.5°.

*o-Tolyl ester*: needles from EtOH. M.p. 120°.

*Anhydride*: yellow cryst. from Me<sub>2</sub>CO. M.p. 182°.

*Anilide*: yellow needles. M.p. 167–8°.

*p-Toluidide*: m.p. 181–2°.

*Trans*:

Yellow prisms or needles from EtOH. M.p. 213–14°. Mod. sol. hot EtOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. NaHg → 4-amino-α-phenylhydrocinnamic acid.

*Me ester*: yellow prisms from EtOH. M.p. 141–2°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Less sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O, pet. ether.

*Phenyl ester*: C<sub>21</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 345. Yellow cryst. from EtOH. M.p. 175–6°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

*o-Tolyl ester*: yellow needles from EtOH. M.p. 128–9°.

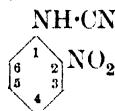
*1-Naphthyl ester*: C<sub>35</sub>H<sub>17</sub>O<sub>4</sub>N. MW, 395. Needles from EtOH. M.p. 126–7°.

*Nitrile*: orange-red powder. M.p. 117–18°.

*Anhydride*: yellow cryst. from Me<sub>2</sub>CO. M.p. 162°. Mod. sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, Et<sub>2</sub>O.

See previous references.

**o-Nitrophenylcyanamide** (2-Nitro-N-cyanoaniline, o-nitrophenylcarbamic nitrile)



C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>N<sub>3</sub> MW, 163

Pale yellow needles. M.p. 152° (146°). Sol. EtOH. Mod. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Sol. alkalis with reddish-brown col.

*N-Benzoyl*: needles from 50% EtOH. M.p. 105°. Sol. EtOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

Pierron, *Bull. soc. chim.*, 1905, **33**, 70.

Arndt, *Ber.*, 1913, **46**, 3528.

Arndt, Rosenau, *Ber.*, 1917, **50**, 1256.

**m-Nitrophenylcyanamide** (3-Nitro-N-cyanoaniline, m-nitrophenylcarbamic nitrile).

Pale yellow needles. M.p. 133–4° (130°). Sol. EtOH. Less sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Sol. alkalis with orange-yellow col.

*N-Benzoyl*: plates. M.p. 109°.

Pierron, *Bull. soc. chim.*, 1905, **33**, 72.

Johnson, Cramer, *J. Am. Chem. Soc.*, 1903, **25**, 491.

Dict. of Org. Comp.—III.

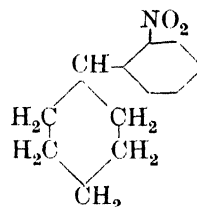
**p-Nitrophenylcyanamide** (4-Nitro-N-cyanoaniline, p-nitrophenylcarbamic nitrile).

Pale yellow needles. M.p. 180°. Sol. EtOH. Less sol. Et<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>.

*N-Benzoyl*: plates from EtOH. M.p. 131°.

Pierron, *Bull. soc. chim.*, 1905, **33**, 73.

**o-Nitrophenylcyclohexane**



C<sub>12</sub>H<sub>15</sub>O<sub>2</sub>N MW, 205

M.p. 45°. B.p. 174°/16 mm., 113°/0.5 mm. D<sub>4</sub><sup>25</sup> 1.111. n<sub>D</sub><sup>25</sup> 1.5472.

Mayes, Turner, *J. Chem. Soc.*, 1929, 503.

Neunhoeffer, *J. prakt. Chem.*, 1932, **133**, 95.

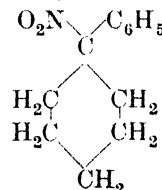
**p-Nitrophenylcyclohexane.**

Yellow plates from EtOH. M.p. 58.5°. B.p. 210°/25 mm., 198°/16 mm. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH.

See first reference above and also

Kurssanow, *Ann.*, 1901, **318**, 321.

**1-Nitro-1-phenylcyclohexane**

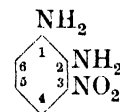


C<sub>12</sub>H<sub>15</sub>O<sub>2</sub>N MW, 205

Needles. M.p. 54.5–56°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>.

Kurssanow, *Chem. Zentr.*, 1907, I, 1744.

**3-Nitro-o-phenylenediamine**



C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub> MW, 153

Dark red needles from EtOH.Aq. M.p. 158–9°. Spar. sol. H<sub>2</sub>O. HNO<sub>2</sub> → 4-nitro-benzotriazole. Acetic anhydride → 4-nitro-2-methylbenzimidazole.

*2-N-Di-Me*: 6-nitro-2-amino-N-dimethylaniline. C<sub>8</sub>H<sub>11</sub>O<sub>2</sub>N<sub>3</sub>. MW, 181. Dark red mass. *1-N-Benzoyl*: yellow needles from EtOH. M.p. 114°.

2-*N*-Phenyl: see 6-Nitro-2-aminodiphenylamine.

*Benzoyl*: dark yellow needles from EtOH. M.p. 206°.

Borsche, Rantschegg, *Ann.*, 1911, **379**, 163.

#### 4-Nitro-*o*-phenylenediamine.

Dark red needles. M.p. 198° (195°).  $\text{HNO}_2 \rightarrow$  5-nitrobenztriazole.

1-*N*-Me: 4-nitro-2-amino-*N*-methylaniline.  $\text{C}_8\text{H}_9\text{O}_2\text{N}_3$ . MW, 167. Reddish-brown needles with blue reflex. M.p. 177-8°.

1:2-*N*-Di-Me:  $\text{C}_8\text{H}_{11}\text{O}_2\text{N}_3$ . MW, 181. Red prisms. M.p. 172°. Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ .

1-*N*-Di-Me: 4-nitro-2-amino-*N*-dimethylaniline. Orange-yellow needles from  $\text{H}_2\text{O}$ . M.p. 63°. Very sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ .

1-*N*-Phenyl: see 4-Nitro-2-aminodiphenylamine.

1-*N*- $\alpha$ -Naphthyl:  $\text{C}_{16}\text{H}_{13}\text{O}_2\text{N}_3$ . MW, 279. Dark yellow needles from AcOH. Very sol. EtOH,  $\text{CHCl}_3$ , AcOH,  $\text{Me}_2\text{CO}$ . Mod. sol.  $\text{Et}_2\text{O}$ .

1-*N*- $\beta$ -Naphthyl: brown needles with green reflex from EtOH.Aq., red prisms from EtOH. M.p. 195°. Very sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ , AcOH. Mod. sol. EtOH,  $\text{C}_6\text{H}_6$ . Insol. ligroin. Brown form  $\rightarrow$  red at 150°. 2-*N*-Acetyl: orange-red needles from boiling EtOH. M.p. 200°. Sol. EtOH,  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ , AcOH. Spar. sol.  $\text{C}_6\text{H}_6$ . Insol. ligroin. 2-*N*-Benzoyl: orange-yellow needles. M.p. 217-18°. Spar. sol. EtOH,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin.

1-*N*-*o*-Tolyl:  $\text{C}_{13}\text{H}_{13}\text{O}_2\text{N}_3$ . MW, 243. Dark red plates or needles from EtOH.Aq. M.p. 121°. Weak base. 2-*N*-Benzoyl: golden-yellow needles from EtOH. M.p. 164-5°.

1-*N*-*p*-Tolyl: m.p. 155-6°. 2-*N*-Benzoyl: yellow needles from EtOH. M.p. 210-11°. Sol. warm EtOH. Spar. sol.  $\text{C}_6\text{H}_6$ .

1-*N*-Acetyl: 4-nitro-2-aminoacetanilide. Cryst. from EtOH. M.p. 205°.

2-*N*-Acetyl: 5-nitro-2-aminoacetanilide. Cryst. from EtOH. M.p. 195°.

1:2-*N*-Diacetyl: prisms from AcOH.Aq. M.p. 255° (227°). Insol. cold EtOH.

8:2-*N*-Dibenzoyl: needles from EtOH. M.p. 235-6°. Sol. EtOH,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ , ligroin.

Heim, *Ber.*, 1888, **21**, 2305.

Brand, *J. prakt. Chem.*, 1906, **74**, 470.

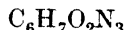
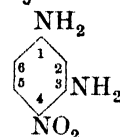
Kehrmann, Messinger, *J. prakt. Chem.*, 1892, **46**, 573.

Fischer, Hess, *Ber.*, 1903, **36**, 3969.

Muttele, *Ann. chim. phys.*, 1898, **14**, 401.

Phillips, *J. Chem. Soc.*, 1928, 174.

#### 4-Nitro-*m*-phenylenediamine



MW, 153

Yellowish-red prisms with blue lustre from  $\text{H}_2\text{O}$ . M.p. 161° (157°). Sol. EtOH,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$ . Decomp. by hot KOH.

1-*N*-Di-Me: 4-nitro-3-amino-*N*-dimethylaniline.  $\text{C}_8\text{H}_{11}\text{O}_2\text{N}_3$ . MW, 181. Cryst. M.p. 135°. 3-*N*-Et:  $\text{C}_{10}\text{H}_{15}\text{O}_2\text{N}_3$ . MW, 209. Orange cryst. M.p. 98°. 3-*N*-Di-Et: orange cryst. M.p. 63-4°.

1:1:3-*N*-Tri-Me:  $\text{C}_9\text{H}_{13}\text{O}_2\text{N}_3$ . MW, 195. Orange needles from MeOH. M.p. 117°.

*N*-Tetra-Me:  $\text{C}_{10}\text{H}_{15}\text{O}_2\text{N}_3$ . MW, 209. Red plates. M.p. 81°.

1-*N*-Di-Et: 4-nitro-3-amino-*N*-diethylaniline.  $\text{C}_{10}\text{H}_{15}\text{O}_2\text{N}_3$ . MW, 209. Yellow cryst. M.p. 139°. 3-*N*-Me:  $\text{C}_{11}\text{H}_{17}\text{O}_2\text{N}_3$ . MW, 223. Yellow cryst. M.p. 96-7°. 3-*N*-Et:  $\text{C}_{12}\text{H}_{19}\text{O}_2\text{N}_3$ . MW, 237. Yellow needles. M.p. 78-5°.

1-*N*-Acetyl: 4-nitro-3-aminoacetanilide. Red prisms from AcOH. M.p. 200°.

1:3-*N*-Diacetyl: needles from EtOH or AcOH.Aq. M.p. 246°. Sol.  $\text{Et}_2\text{O}$ , AcOH. Sol. 100 parts EtOH. Insol.  $\text{H}_2\text{O}$ .

1:3-*N*-Dibenzoyl: yellow needles from AcOH. M.p. 222°. Spar. sol. EtOH.

1:3-*N*-Di-*p*-toluenesulphonyl: brownish-yellow cryst. M.p. 169°. Sol. alkalis. Spar. sol.  $\text{H}_2\text{O}$ .

Gallinek, *Ber.*, 1897, **30**, 1912.

A.G.F.A., D.R.P., 130,438, (*Chem. Zentr.*, 1902, I, 1083).

Morgan, Wootton, *J. Chem. Soc.*, 1905, **87**, 941.

Romburgh, *Rec. trav. chim.*, 1923, **42**, 804.

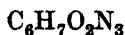
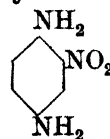
Forster, Coulson, *J. Chem. Soc.*, 1922, **121**, 1996.

#### 5-Nitro-*m*-phenylenediamine.

Purplish-red cryst. from  $\text{H}_2\text{O}$ . M.p. 140-1°. 1:3-*N*-Diacetyl: yellowish needles. M.p. above 270° decomp. Sol.  $\text{PhNO}_2$ . Insol. other solvents.

Flürscheim, *J. prakt. Chem.*, 1905, **71**, 538.

#### 2-Nitro-*p*-phenylenediamine



MW, 153

Black needles with strong green reflex from  $\text{H}_2\text{O}$ . M.p.  $137^\circ$  ( $134-5^\circ$ ).

1-N-Me: 2-nitro-4-amino-N-methylaniline.  $\text{C}_7\text{H}_9\text{O}_2\text{N}_3$ . MW, 167. Black prisms from  $\text{H}_2\text{O}$ . M.p.  $109-10^\circ$ . Sol. dil. acids with yellowish-red col.

1-N-Di-Me: 2-nitro-4-amino-N-dimethylaniline.  $\text{C}_8\text{H}_{11}\text{O}_2\text{N}_3$ . MW, 181. 4-N-Acetyl: 3-nitro-p-dimethylaminoacetanilide. Maroon cryst. M.p.  $132^\circ$ . Hydrochloride of acetyl: m.p.  $180^\circ$  decomp. Picrate of N-acetyl: orange plates. M.p.  $172^\circ$ .

4-N-Di-Me: 3-nitro-4-amino-N-dimethylaniline. Red needles. M.p.  $112^\circ$ . Picrate: greenish-yellow prisms. M.p.  $205^\circ$ . 1-N-Acetyl: 2-nitro-p-dimethylaminoacetanilide. Red needles. M.p.  $116^\circ$ . Picrate of N-acetyl: lemon-yellow plates. M.p.  $185^\circ$ .

1-N-Phenyl: see 2-Nitro-4-aminodiphenylamine.

N-Tetraphenyl:  $\text{C}_{30}\text{H}_{23}\text{O}_2\text{N}_3$ . MW, 457. Red needles from AcOH. M.p.  $167-8^\circ$ .

1-N-Acetyl: 2-nitro-4-aminoacetanilide. Red needles from  $\text{H}_2\text{O}$ . M.p.  $162.5^\circ$ .

4-N-Acetyl: 3-nitro-4-aminoacetanilide. Red needles or leaflets from  $\text{H}_2\text{O}$ . M.p.  $189^\circ$ .

1:4-N-Diacetyl: yellow needles from  $\text{H}_2\text{O}$ . M.p.  $186^\circ$ .

4-N-Benzoyl: yellowish-red needles. M.p.  $236^\circ$ .

1:4-N-Di-p-nitrobenzoyl: yellow leaflets from Py-EtOH. Does not melt below  $305^\circ$ .

Kym, *Ber.*, 1911, **44**, 2923.

Chazel, *Ber.*, 1907, **40**, 3183.

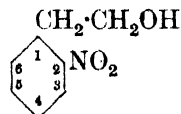
Brand, *J. prakt. Chem.*, 1906, **74**, 470.

Hodgson, Crook, *J. Chem. Soc.*, 1932, 2976.

Phillips, *J. Chem. Soc.*, 1928, 172.

Haeussermann, Bauer, *Ber.*, 1899, **32**, 1913.

**o-Nitrophenylethyl Alcohol** (o-Nitro- $\beta$ -hydroxyethylbenzene)



$\text{C}_8\text{H}_9\text{O}_3\text{N}$

MW, 167

Colourless oil. B.p.  $144-7^\circ/1.3$  mm.  $D^{25}_{20}$  1.5620. Non-volatile in steam.

Benzoyl: plates from pet. ether. M.p.  $55^\circ$ .

Sabetay, Bléger, Lestrangé, *Bull. soc. chim.*, 1931, **49**, 3.

**p-Nitrophenylethyl Alcohol** (p-Nitro- $\beta$ -hydroxyethylbenzene).

Yellowish needles. M.p.  $64^\circ$ . B.p.  $177^\circ/16$  mm.

Acetyl: b.p.  $189^\circ/16$  mm.

m-Nitrobenzoyl: m.p.  $64-5^\circ$ .

Phenylurethane: m.p.  $127-8^\circ$ .

Pishchimuka, *Chem. Abstracts*, 1917, **11**, 451.

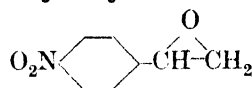
v. Braun, Bartsch, *Ber.*, 1913, **46**, 3053.

Ehrlich, Pitschimuka, *Ber.*, 1912, **45**, 2432.

**o-Nitrophenylethylene oxide.**

See Nitraldin.

**p-Nitrophenylethylene oxide**



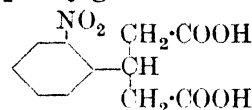
$\text{C}_8\text{H}_7\text{O}_3\text{N}$

MW, 165

Prisms from MeOH. M.p.  $84-5^\circ$ .

Arndt, Eistert, Ender, *Ber.*, 1929, **62**, 51.

**2-o-Nitrophenylglutaric Acid**



$\text{C}_{11}\text{H}_{11}\text{O}_6\text{N}$

MW, 253

Prisms from  $\text{H}_2\text{O}$ . M.p.  $205^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH, AcOH, AcOEt. Insol. Et<sub>2</sub>O,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether.

Di-Me ester:  $\text{C}_{13}\text{H}_{15}\text{O}_6\text{N}$ . MW, 281. Prisms from Et<sub>2</sub>O. M.p.  $65.5^\circ$ . Very sol. AcOH, AcOEt,  $\text{C}_6\text{H}_6$ .

Anhydride:  $\text{C}_{22}\text{H}_{20}\text{O}_{11}\text{N}_2$ . MW, 488. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $130-1^\circ$ .

Schroeter, Meerwein, *Ber.*, 1903, **36**, 2672; 1907, **40**, 1586.

**2-m-Nitrophenylglutaric Acid.**

Needles from EtOH.Aq. M.p.  $205-6^\circ$ . Sol. EtOH, Et<sub>2</sub>O. Mod. sol. AcOH. Spar. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin, pet. ether.

Anhydride: cryst. M.p.  $170.5^\circ$ . Insol.  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether.

Avery, Gere, *Am. Chem. J.*, 1902, **28**, 51.

Knoevenagel, Schürenberg, *Ann.*, 1898, **303**, 235.

See also previous references.

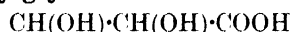
**2-p-Nitrophenylglutaric Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $240^\circ$  ( $235^\circ$ ). Sol. EtOH, AcOH. Spar. sol.  $\text{H}_2\text{O}$ . Insol. Et<sub>2</sub>O,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin.

Di-Me ester: plates from EtOH. M.p.  $65^\circ$  ( $62^\circ$ ). Very sol. EtOH. Insol.  $\text{H}_2\text{O}$ .

*Anhydride*: pale yellow cryst. from AcOEt. M.p. 122-5°. Spar. sol. hot C<sub>6</sub>H<sub>6</sub>.

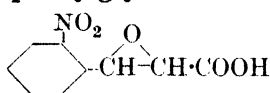
Avery, Beans, *Am. Chem. J.*, 1902, **28**, 58.  
See also previous references.

**2-*p*-Nitrophenylglyceric Acid**C<sub>9</sub>H<sub>9</sub>O<sub>6</sub>N

MW, 227

Yellow leaflets from H<sub>2</sub>O. M.p. 167-8°. Sol. EtOH, hot H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O.

Lipp, *Ber.*, 1886, **19**, 2645.

**2-*o*-Nitrophenylglycidic Acid**C<sub>9</sub>H<sub>7</sub>O<sub>5</sub>N

MW, 209

Plates from C<sub>6</sub>H<sub>6</sub>, m.p. 124-5-125°; needles + 1H<sub>2</sub>O from H<sub>2</sub>O, m.p. 94°. Sol. EtOH. Et<sub>2</sub>O, hot H<sub>2</sub>O, hot C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. Heat → indigo. Conc. H<sub>2</sub>SO<sub>4</sub> → red col.

*Me ester*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Needles from ligroin. M.p. 65°.

Einhorn, Gernsheim, *Ann.*, 1895, **284**, 135.

Lipp, *Ber.*, 1886, **19**, 2649.

**2-*p*-Nitrophenylglycidic Acid.**

*Cis*:

Cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 124-5°.

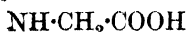
*Trans*:

Cryst. from EtOH. M.p. 186-8° decomp. Spar. sol. boiling H<sub>2</sub>O, cold EtOH.

Kleucker, *Ber.*, 1922, **55**, 1646.

Lipp, *Ber.*, 1886, **19**, 2644.

Erlenmeyer, *Ber.*, 1881, **14**, 1868.

***o*-Nitrophenylglycine**C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub>

MW, 196

Dark red prisms from EtOH. M.p. 192-3° decomp. Sol. hot EtOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O.

Plöchl, *Ber.*, 1886, **19**, 7.

Borsche, Titsingh, *Ber.*, 1907, **40**, 5016.

***m*-Nitrophenylglycine.**

Yellow prisms from H<sub>2</sub>O. M.p. 158-9°. Sol. EtOH, pet. ether.

*Et ester*: C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub>. MW, 224. Cryst. from EtOH. M.p. 84°.

*m*-Nitroanilide: cryst. M.p. 201-2°.

Borsche, Titsingh, *Ber.*, 1907, **40**, 5015.

Deutsch, *J. prakt. Chem.*, 1907, **76**, 352.

***p*-Nitrophenylglycine.**

Yellow cryst. from H<sub>2</sub>O. M.p. 225° decomp.

*N*-Formyl: brownish-yellow plates from AcOH. M.p. 159-60° decomp. Very sol. hot AcOH. Sol. boiling EtOH. Insol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*N*-Acetyl: brownish leaflets from AcOH. M.p. 191-2°. Very sol. hot AcOH. Sol. hot EtOH. Insol. cold H<sub>2</sub>O.

*p*-Nitroanilide: m.p. 260°.

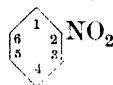
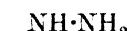
M.L.B., D.R.P., 88,433.

Badische, D.R.P., 152,012, (*Chem. Zentr.*, 1904, II, 70).

Borsche, Titsingh, *Ber.*, 1907, **40**, 5016.

**Nitrophenylhydracrylic Acid.**

See Nitro-β-hydroxyhydrocinnamic Acid.

***o*-Nitrophenylhydrazine**C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub>

MW, 153

Deep red needles from C<sub>6</sub>H<sub>6</sub>. M.p. 90°. Sol. hot H<sub>2</sub>O. Spar. sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

*Formyl deriv.*: needles from EtOH. M.p. 177°.

*Acetyl deriv.*: yellow needles from EtOH. M.p. 140-1°.

*Diacetyl deriv.*: prisms from EtOH. M.p. 57-8°.

*Benzoyl deriv.*: yellow needles from EtOH. M.p. 166°.

Bischler, *Ber.*, 1889, **22**, 2801.

***m*-Nitrophenylhydrazine.**

Yellow needles from EtOH. M.p. 93°. Sol. AcOH, CHCl<sub>3</sub>. Spar. sol. EtOH, C<sub>6</sub>H<sub>6</sub>, boiling H<sub>2</sub>O.

*Acetyl deriv.*: yellow leaflets from H<sub>2</sub>O. M.p. 145°.

*Diacetyl deriv.*: plates from AcOH. M.p. 150°.

*Benzoyl deriv.*: prismatic needles from EtOH.Aq. M.p. 151°.

*Dibenzoyl deriv.*: yellow leaflets from AcOH.Aq. M.p. 153°.

Bischler, Brodsky, *Ber.*, 1889, **22**, 2809.

Hantzsch, Borghaus, *Ber.*, 1897, **30**, 91.

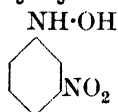
**p-Nitrophenylhydrazine.**

Orange-red leaflets and needles from boiling EtOH. M.p. 157° decomp. Sol. warm EtOH. Mod. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

*Picrate*: red needles from H<sub>2</sub>O. M.p. 119–20°. Sol. hot H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin.

Bamberger, Kraus, *Ber.*, 1896, **29**, 1834.

**N-m-Nitrophenylhydroxylamine**



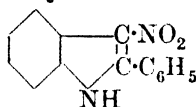
C<sub>6</sub>H<sub>6</sub>O<sub>3</sub>N<sub>2</sub>

MW, 154

Yellow cryst. from hot C<sub>6</sub>H<sub>6</sub>. M.p. 118–19°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold H<sub>2</sub>O. Reduces NH<sub>3</sub>·AgNO<sub>3</sub> and Fehling's. Alc. FeCl<sub>3</sub> → 3-nitrosanitrobenzene. Warm 60% H<sub>2</sub>SO<sub>4</sub> → 2-nitro-p-aminophenol.

Brand, *J. prakt. Chem.*, 1906, **74**, 464; *Ber.*, 1905, **38**, 4010.

**3-Nitro-2-phenylindole**



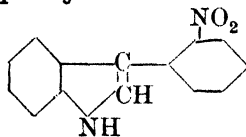
C<sub>14</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

MW, 238

Yellow cryst. from AcOH or EtOH. M.p. 238–9°. Alk. KMnO<sub>4</sub> → N-benzoylanthranilic acid.

Angeli, Angelico, *Gazz. chim. ital.*, 1900, **30**, ii, 275.

**3-o-Nitrophenylindole**



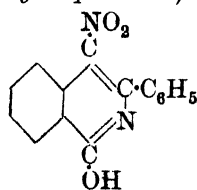
C<sub>14</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

MW, 238

Orange prismatic needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 119°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Mod. sol. pet. ether. Ehrlich's reagent → pink col.

Kermack, Slater, *J. Chem. Soc.*, 1928, 38.

**4-Nitro-3-phenylisocarbostyryl (4-Nitro-1-hydroxy-3-phenylisoquinoline)**



C<sub>15</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>

MW, 266

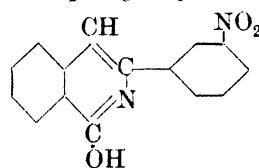
Yellow cryst. from EtOH. M.p. about 245° decomp. Sol. boiling AcOH. Spar. sol. EtOH, Et<sub>2</sub>O, cold C<sub>6</sub>H<sub>6</sub>. Very spar. sol. CS<sub>2</sub>, ligroin.

*Me ether*: C<sub>16</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 280. Yellow cryst. from EtOH. M.p. 167–9°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, hot AcOH. Mod. sol. hot EtOH. Spar. sol. CS<sub>2</sub>, ligroin.

Gabriel, *Ber.*, 1886, **19**, 831.

Wölbling, *Ber.*, 1905, **38**, 3850.

**3-m-Nitrophenylisocarbostyryl (1-Hydroxy-3-m-nitrophenylisoquinoline)**



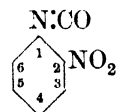
C<sub>15</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>

MW, 266

Cryst. from PhNO<sub>2</sub>. M.p. 298–300°. Sol. AcOH. Insol. EtOH, Et<sub>2</sub>O.

Harper, *Ber.*, 1896, **29**, 2545.

**o-Nitrophenyl isocyanate (o-Nitrophenyl-carbonimide)**



C<sub>7</sub>H<sub>4</sub>O<sub>3</sub>N<sub>2</sub>

MW, 164

Needles from pet. ether. M.p. 41° (37–8°). Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

Hoeke, *Rec. trav. chim.*, 1935, **54**, 505.

**m-Nitrophenyl isocyanate (m-Nitrophenyl-carbonimide).**

Leaflets. M.p. 51°. Sol. C<sub>6</sub>H<sub>6</sub>, Et<sub>2</sub>O, CHCl<sub>3</sub>, toluene, boiling ligroin.

Folin, *Am. Chem. J.*, 1897, **19**, 339.

Hoeke, *Rec. trav. chim.*, 1935, **54**, 505.

**p-Nitrophenyl isocyanate (p-Nitrophenyl-carbonimide).**

Needles. M.p. 57°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, toluene, boiling ligroin.

See last reference above.

**o-Nitrophenyl isothiocyanate (o-Nitro-phenylthiocarbonimide)**



C<sub>7</sub>H<sub>4</sub>O<sub>2</sub>N<sub>2</sub>S

MW, 180

Yellow plates from Me<sub>2</sub>CO.Aq. M.p. 74°.

Dyson, *J. Chem. Soc.*, 1934, 176.

**m-Nitrophenyl isothiocyanate** (*m-Nitrophenylthiocarbonimide*).

Needles from AcOH. M.p. 60.5°. B.p. 275–80° decomp. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Combines readily with bases and alcohols.

Steudemann, *Ber.*, 1883, **16**, 549, 2334.

**p-Nitrophenyl isothiocyanate** (*p-Nitrophenylthiocarbonimide*).

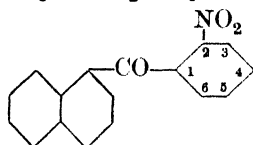
Pale yellow needles from AcOH. M.p. 112–13°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, warm AcOH.

Jacobson, Klein, *Ber.*, 1893, **26**, 2369.

**β-2-Nitrophenyl-lactic Acid.**

See *o*-Nitro-α-hydroxyhydrocinnamic Acid.

**o-Nitrophenyl 1-naphthyl Ketone**



C<sub>17</sub>H<sub>11</sub>O<sub>3</sub>N

MW, 277

Orange-yellow prisms. M.p. 122°.

Berlingozzi, *Chem. Zentr.*, 1934, **II**, 601.

**m-Nitrophenyl 1-naphthyl Ketone.**

Reddish-yellow cryst. M.p. 117°.

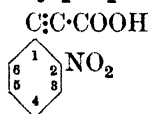
See previous reference.

**p-Nitrophenyl 1-naphthyl Ketone.**

Yellow micro-cryst. M.p. 95°.

See previous reference.

**o-Nitrophenylpropionic Acid**



C<sub>9</sub>H<sub>5</sub>O<sub>4</sub>N

MW, 191

Needles or leaflets from H<sub>2</sub>O. M.p. 157° decomp. Sol. hot H<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>. Insol. CS<sub>2</sub>, ligroin.  $k = 1.06 \times 10^{-2}$  at 25°. NH<sub>3</sub>.Aq. + FeSO<sub>4</sub> → *o*-aminophenylpropionic acid. Hot alkalis → isatin. Heat with H<sub>2</sub>O → *o*-nitrophenylacetylene.

*Me ester*: C<sub>10</sub>H<sub>7</sub>O<sub>4</sub>N. MW, 205. Yellowish leaflets from CCl<sub>4</sub>. M.p. 87–8°. Very sol. MeOH, CHCl<sub>3</sub>.

*Et ester*: C<sub>11</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 219. Pale yellow leaflets from EtOH or Et<sub>2</sub>O. M.p. 62°. (NH<sub>4</sub>)<sub>2</sub>S → indoxyllic acid ethyl ester.

*Amide*: C<sub>9</sub>H<sub>6</sub>O<sub>3</sub>N<sub>2</sub>. MW, 190. Plates from H<sub>2</sub>O. M.p. 159°.

Baeyer, *Ber.*, 1880, **13**, 2258.

Pfeiffer, *Ann.*, 1916, **411**, 148.

Rinkes, *Rec. trav. chim.*, 1929, **48**, 960.

**m-Nitrophenylpropionic Acid.**

Needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 143°. Sol. EtOH, Et<sub>2</sub>O, AcOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, hot H<sub>2</sub>O. Insol. ligroin. Heat with H<sub>2</sub>O → *m*-nitrophenylacetylene.

Wollring, *Ber.*, 1914, **47**, 111.

Reich, Koehler, *Ber.*, 1913, **46**, 3735.

**p-Nitrophenylpropionic Acid.**

Needles from EtOH or Et<sub>2</sub>O. M.p. 181°. Sol. hot EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. H<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Very spar. sol. CS<sub>2</sub>. Insol. pet. ether. Sol. cold conc. H<sub>2</sub>SO<sub>4</sub>. Heat with H<sub>2</sub>O → *p*-nitrophenylacetylene.

*Et ester*: needles from Et<sub>2</sub>O. M.p. 126°.

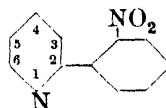
Perkin, Bellenot, *J. Chem. Soc.*, 1886, **49**, 441.

Reich, *Compt. rend.*, 1916, **162**, 129.

**Nitrophenylpropionic Acid.**

See Nitrohydratropic Acid and Nitrohydrocinnamic Acid.

**2-o-Nitrophenylpyridine**



C<sub>11</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub>

MW, 200

Tablets. M.p. 60–1°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Forsyth, Pyman, *J. Chem. Soc.*, 1926, 2919.

**2-m-Nitrophenylpyridine.**

Pale yellow needles from EtOH. M.p. 73–4°. Sol. Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, EtOH.

*B, HNO<sub>3</sub>*: needles from 5*N*/HNO<sub>3</sub>. M.p. 193°.

See previous reference.

**2-p-Nitrophenylpyridine.**

Needles from EtOH. M.p. 130.5–131.5°. Sol. CHCl<sub>3</sub>. Spar. sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Insol. H<sub>2</sub>O.

*B, HCl*: needles from 5*N*/HCl. M.p. 185–6°.

*Picrate*: m.p. 131–131.5°.

Forsyth, Pyman, *J. Chem. Soc.*, 1926, 2916.

Tschitschibabin, Schemjakina, *Chem. Zentr.*, 1923, **III**, 1024.

**3-p-Nitrophenylpyridine.**

Buff needles from EtOH. M.p. 148–9°. Sol. CHCl<sub>3</sub>. Spar. sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Insol. H<sub>2</sub>O.

*B, HNO<sub>3</sub>*: needles from *N/HNO<sub>3</sub>*. M.p. 198°. Forsyth, Pyman, *J. Chem. Soc.*, 1926, 2920.

**4-*o*-Nitrophenylpyridine.**

Plates from Et<sub>2</sub>O. M.p. 51–2°. *B, HNO<sub>3</sub>*: prisms from 5*N/HNO<sub>3</sub>*. M.p. 178–9°. Forsyth, Pyman, *J. Chem. Soc.*, 1926, 2924.

**4-*m*-Nitrophenylpyridine.**

Needles from Me<sub>2</sub>CO. M.p. 109–10°. Sol. EtOH, Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O. *B, HNO<sub>3</sub>*: needles from 5*N/HNO<sub>3</sub>*. M.p. 222° decomp.

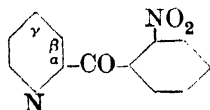
See previous reference.

**4-*p*-Nitrophenylpyridine.**

Prisms from Me<sub>2</sub>CO. M.p. 123–4°. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O. *B, HCl*: prisms from 5*N/HCl*. M.p. 255°.

Tschitschibabin, Shemyakina, *Chem. Zentr.*, 1923, III, 1024.

***o*-Nitrophenyl α-pyridyl Ketone (2-*o*-Nitrobenzoylpyridine)**



C<sub>12</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub> MW, 228  
Needles from H<sub>2</sub>O. M.p. 118°.

Wilson, *J. Chem. Soc.*, 1931, 1937.

***m*-Nitrophenyl α-pyridyl Ketone (2-*m*-Nitrobenzoylpyridine).**

Prisms from EtOH, needles from H<sub>2</sub>O. M.p. 122°.

Bryans, Pyman, *J. Chem. Soc.*, 1929, 551.

***p*-Nitrophenyl α-pyridyl Ketone (2-*p*-Nitrobenzoylpyridine).**

Needles from H<sub>2</sub>O. M.p. 110° (100°). Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOEt, C<sub>6</sub>H<sub>6</sub>, ligroin. *B, HCl*: needles from EtOH. M.p. 187° (173°).

*Phenyldiazotone*: yellow cryst. from EtOH. M.p. 171°.

Tschitschibabin, Kuindshi, Benewolenskaja, *Ber.*, 1925, 58, 1584.

Koenigs, Mensching, Kirsch, *Ber.*, 1926, 59, 1719.

Bryans, Pyman, *J. Chem. Soc.*, 1929, 551.

***p*-Nitrophenyl β-pyridyl Ketone (3-*p*-Nitrobenzoylpyridine).**

Needles from H<sub>2</sub>O. M.p. 106°.

*Picrate*: plates from H<sub>2</sub>O. M.p. 185–7°.

Bryans, Pyman, *J. Chem. Soc.*, 1929, 552.

***m*-Nitrophenyl γ-pyridyl Ketone (4-*m*-Nitrobenzoylpyridine).**

Needles from H<sub>2</sub>O. M.p. 129°.

See previous reference.

***p*-Nitrophenyl γ-pyridyl Ketone (4-*p*-Nitrobenzoylpyridine).**

Needles from H<sub>2</sub>O. M.p. 123–4°. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

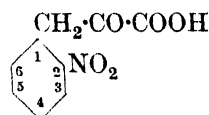
*B, HCl*: m.p. 202°.

*Phenyldiazotone*: m.p. 226°.

Tschitschibabin, Kuindshi, Benewolenskaja, *Ber.*, 1925, 58, 1587.

Koenigs, Mensching, Kirsch, *Ber.*, 1926, 59, 1722.

***o*-Nitrophenylpyruvic Acid**



C<sub>9</sub>H<sub>7</sub>O<sub>5</sub>N

MW, 209

*Enol form*: see 2-Nitro-α-hydroxycinnamic Acid.

Yellowish needles or leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 121°. Very sol. EtOH, Et<sub>2</sub>O, AcOH. Mod. sol. hot H<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Almost insol. ligroin. FeCl<sub>3</sub> → olive-green col.

*Me ester*: C<sub>10</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 223. Yellow needles. M.p. 83–7°.

*Et ester*: C<sub>11</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 237. Needles or prisms from ligroin. M.p. 46–7°. Very sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, AcOH. Spar. sol. ligroin. FeCl<sub>3</sub> gives no immediate col. → green on standing. Gives no col. with Cu acetate. *Oxime*: needles from EtOH. M.p. 121–2°. *Phenyldiazotone*: yellow needles from EtOH. M.p. 103–5°. FeCl<sub>3</sub> in conc. H<sub>2</sub>SO<sub>4</sub> → purplish-red col. *Diphenyldiazotone*: needles. M.p. 107°. *Anil*: red prisms from EtOH. M.p. 92°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. ligroin. *o-Tolanil*: orange cryst. M.p. 77–8°. *m-Tolanil*: orange cryst. M.p. 138°. *p-Tolanil*: dark red cryst. from EtOH. M.p. 90–5–91–0°. *p-Bromoanil*: dark red prisms. M.p. 136–7°.

*Amide*: C<sub>9</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub>. MW, 208. Needles from EtOH. M.p. 165–6°. Sol. AcOH with decomp. Mod. sol. hot H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O. *Oxime*: needles. M.p. 161° decomp. Sol.



Me<sub>2</sub>CO, AcOH. Mod. sol. EtOH, Et<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether.

unsym.-*Diphenylhydrazone*: yellow needles from EtOH. M.p. 125°.

unsym.-*Methylphenylhydrazone*: yellow needles from EtOH. M.p. 110°.

Reissert, D.R.P., 92,794; *Ber.*, 1897, 30, 1036; 1908, 41, 3813.

Wislicenus, Thoma, *Ann.*, 1924, 436, 45.

**p-Nitrophenylpyruvic Acid.**

*Enol-form*: see 4-Nitro- $\alpha$ -hydroxycinnamic Acid.

Orange yellow cryst. + AcOH from AcOH. M.p. 194°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Mod. sol. AcOH. Spar. sol. H<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. FeCl<sub>3</sub>  $\rightarrow$  bluish-green col.

*Me ester*: prisms from MeOH-Me<sub>2</sub>CO. M.p. 149°. *Oxime*: pale yellow cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 172-3°. Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, EtOH. *Phenylhydrazone*: yellowish-green cryst. from MeOH. M.p. 136-45°.

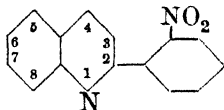
*Py salt*: yellowish-green needles from Py. M.p. 150°. Sol. Na<sub>2</sub>CO<sub>3</sub> with red col.

Reissert, *Ber.*, 1897, 30, 1047.

Wislicenus, Schultz, *Ann.*, 1924, 436, 57.

See also first reference above.

**2-o-Nitrophenylquinoline**



C<sub>15</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

MW, 250

Needles from MeOH.Aq. M.p. 121-3°.

Le Fèvre, Mathur, *J. Chem. Soc.*, 1930, 2240.

**2-m-Nitrophenylquinoline.**

Pale yellow needles from EtOH. M.p. 123-4°.

*Methochloride*: needles from EtOH-Et<sub>2</sub>O. M.p. 204-5°. Sol. H<sub>2</sub>O, MeOH, EtOH. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

*Methopicate*: cryst. from EtOH. M.p. 181-2°.

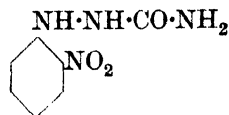
Le Fèvre, Mathur, *J. Chem. Soc.*, 1930, 2239.

**2-p-Nitrophenylquinoline.**

Pale yellow needles from MeOH.Aq. M.p. 129-31°.

Le Fèvre, Mathur, *J. Chem. Soc.*, 1930, 2240.

**1-o-Nitrophenylsemicarbazide**



C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>N<sub>4</sub>

MW, 196

Yellow cryst. from EtOH. M.p. 225° decomp.

Guha, Ghosh, *J. Indian Chem. Soc.*, 1928, 5, 447.

**1-m-Nitrophenylsemicarbazide.**

Needles from EtOH. M.p. 195° decomp. Sol. hot H<sub>2</sub>O, EtOH. Spar. sol. boiling Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

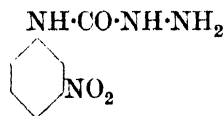
Young, Stockwell, *J. Chem. Soc.*, 1898, 73, 372.

**1-p-Nitrophenylsemicarbazide.**

Yellow needles from H<sub>2</sub>O. M.p. 211-12° decomp. Sol. hot H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Dil. NaOH  $\rightarrow$  orange-red sol.

Hyde, *Ber.*, 1899, 32, 1812.

**4-m-Nitrophenylsemicarbazide**



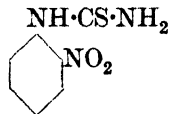
C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>N<sub>4</sub>

MW, 196

Yellow needles from EtOH. M.p. 138-9°. Sol. 4 parts boiling EtOH. Conc. alkalis  $\rightarrow$  red col. Reduces Fehling's and NH<sub>3</sub>.AgNO<sub>3</sub>.

Wheeler, Walker, *J. Am. Chem. Soc.*, 1925, 47, 2794.

**o-Nitrophenylthiourea**



C<sub>7</sub>H<sub>7</sub>O<sub>2</sub>N<sub>3</sub>S

MW, 197

Yellow cryst. from EtOH. M.p. 140-141.5° (136°). Sol. Et<sub>2</sub>O. Sol. alkalis with light red col. Conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  mauve col.

Arndt, Rosenau, *Ber.*, 1917, 50, 1255.

Dyson, George, *J. Chem. Soc.*, 1924, 125, 1707.

**m-Nitrophenylthiourea.**

Yellow cryst. M.p. 120-5° decomp. (157-8°).

See second reference above and also Steudemann, *Ber.*, 1883, 16, 550.

**p-Nitrophenylthiourea.**

Yellow cryst. M.p. 189–90°.

Dyson, George, *J. Chem. Soc.*, 1924, 125, 1707.**Nitrophenyl tolyl Ether.**

See Nitromethyldiphenyl Ether.

**Nitrophenyl tolyl Ketone.**

See Nitromethylbenzophenone.

**Nitrophenyltolylmethane.**

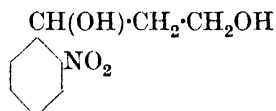
See Nitromethyldiphenylmethane.

**Nitrophenyl tolyl sulphide.**

See Nitromethyldiphenyl sulphide.

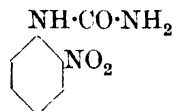
**Nitrophenyl tolyl sulphone.**

See Nitromethyldiphenyl sulphone.

**1-o-Nitrophenyltrimethylene Glycol** (2-Nitro- $\alpha$ -*γ*-dihydroxy-1-propylbenzene) $\text{C}_9\text{H}_{11}\text{O}_4\text{N}$ 

MW, 197

Needles from EtOH. M.p. 108–9°.

Baeyer, Drewsen, *Ber.*, 1882, 15, 2861.**o-Nitrophenylurea** $\text{C}_7\text{H}_7\text{O}_3\text{N}_3$ 

MW, 181

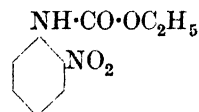
Yellow needles from  $\text{H}_2\text{O}$ . M.p. 183–4° (181°).  
N'-Phenyl: see 2-Nitrocarbanilide.Arndt, *Ber.*, 1913, 46, 3529.Reudler, *Rec. trav. chim.*, 1914, 33, 46.**m-Nitrophenylurea.**Yellow needles or plates from  $\text{H}_2\text{O}$ . M.p. 196°.

N'-Phenyl: see 3-Nitrocarbanilide.

Reudler, *Rec. trav. chim.*, 1914, 33, 46.Pierron, *Bull. soc. chim.*, 1905, 33, 72.**p-Nitrophenylurea.**Prisms from EtOH, needles from EtOH.Aq. M.p. 238°. Sol. EtOH, boiling  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .N'-Me:  $\text{C}_8\text{H}_9\text{O}_3\text{N}_3$ . MW, 195. Yellow needles. M.p. 225–7°. Sol.  $\text{Me}_2\text{CO}$ , hot EtOH, AcOH. Spar. sol. hot  $\text{H}_2\text{O}$ , AcOEt.

N'-Phenyl: see 4-Nitrocarbanilide.

See previous references and also

Scholl, Nyberg, *Ber.*, 1906, 39, 2492.**o-Nitrophenylurethane** (2-Nitrocarbanilic acid ethyl ester) $\text{C}_9\text{H}_{10}\text{O}_4\text{N}_2$ 

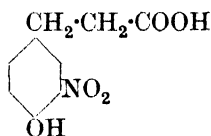
MW, 210

Yellow prisms from pet. ether, needles from EtOH. M.p. 58° (56°). Sol.  $\text{C}_6\text{H}_6$ , ligroin. Spar. sol.  $\text{Et}_2\text{O}$ .Swartz, *Am. Chem. J.*, 1897, 19, 303.Vittenet, *Bull. soc. chim.*, 1899, 21, 588.**m-Nitrophenylurethane** (3-Nitrocarbanilic acid ethyl ester).Pale yellow needles. M.p. 56–7° (65°). Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ .

See previous references and also

Curtius, Struve, Radenhausen, *J. prakt. Chem.*, 1895, 52, 230.**p-Nitrophenylurethane** (4-Nitrocarbanilic acid ethyl ester).Needles from  $\text{C}_6\text{H}_6$ . M.p. 129° (132°). Sol. EtOH,  $\text{C}_6\text{H}_6$ . Insol. boiling ligroin.

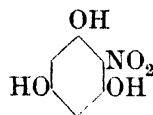
N-Me: m.p. 45°.

Jacobson, Klein, *Ber.*, 1893, 26, 2370.Curtius, Struve, Radenhausen, *J. prakt. Chem.*, 1895, 52, 233.Shriner, Cox, *J. Am. Chem. Soc.*, 1931, 53, 1604.**3-Nitrophenetic Acid** (3-Nitro-p-hydrocoumaric acid, 3-nitro-4-hydroxyhydrocinnamic acid) $\text{C}_9\text{H}_9\text{O}_5\text{N}$ 

MW, 211

Orange-yellow needles from  $\text{H}_2\text{O}$ . M.p. 90–5°. Very sol. EtOH. Spar. sol. boiling  $\text{H}_2\text{O}$ .Me ester:  $\text{C}_{10}\text{H}_{11}\text{O}_5\text{N}$ . MW, 225. Yellow needles from EtOH.Aq. M.p. 64°.Et ester:  $\text{C}_{11}\text{H}_{13}\text{O}_5\text{N}$ . MW, 239. Yellow needles from EtOH.Aq. M.p. 38°.Me ether:  $\text{C}_{10}\text{H}_{11}\text{O}_5\text{N}$ . MW, 225. Pale yellow needles from  $\text{CCl}_4$ . M.p. 128–130·5°.Amide:  $\text{C}_{10}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 224. Pale yellow cryst. M.p. 126·5–127°.Stöhr, *Ann.*, 1884, 225, 92.Bougault, *Ann. chim. phys.*, 1902, 25, 504.Callow, Gulland, Haworth, *J. Chem. Soc.*, 1929, 1452.

**Nitrophloroglucinol** (2-Nitro-1:3:5-tri-hydroxybenzene)



$C_6H_5O_5N$

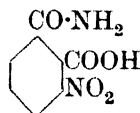
MW, 171

Red leaflets or prisms +  $H_2O$  from  $H_2O$ . M.p.  $189-91^\circ$  ( $186-7^\circ$ ), anhyd.  $205^\circ$ . Sol. AcOH. Mod. sol. EtOH,  $Et_2O$ ,  $Me_2CO$ , AcOEt. Spar. sol.  $H_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol. pet. ether. Sol.  $Na_2CO_3$  with red col.

Leuchs, Geserick, *Ber.*, 1908, **41**, 4182.

Rüdiger, *Arch. Pharm.*, 1914, **252**, 180.

**3-Nitrophthalamic Acid** (3-Nitrophthalic acid 1-amide)



$C_8H_6O_5N_2$

MW, 210

Plates from warm  $H_2O$ . M.p. depends on rate of heating. Starts to melt at  $150-6^\circ$ , re-solidifies and then melts at  $214-15^\circ$ . Sol. hot  $H_2O$ . Mod. sol. EtOH,  $Me_2CO$ , AcOH. Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , ligroin. Above  $160^\circ \rightarrow$  3-nitrophthalimide.

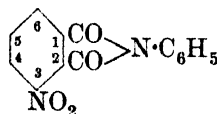
$NH_4$  salt: needles from  $H_2O$ . M.p.  $172^\circ$  decomp. Above m.p.  $\rightarrow$  3-nitrophthalimide.

Chambers, *J. Am. Chem. Soc.*, 1903, **25**, 608.

Kahn, *Ber.*, 1902, **35**, 3862.

Chapman, Stephen, *J. Chem. Soc.*, 1925, 1795.

**3-Nitrophthalanil** (3-Nitrophthalic acid anil, 3-nitro-N-phenylphthalimide)



$C_{14}H_8O_4N_2$

MW, 268

Yellowish needles from EtOH- $Me_2CO$ . M.p.  $138^\circ$  ( $134^\circ$ ). Sol.  $Me_2CO$ . Spar. sol. cold EtOH. Insol.  $H_2O$ .

Bogert, Boroschek, *J. Am. Chem. Soc.*, 1901, **23**, 748.

Chambers, *J. Am. Chem. Soc.*, 1903, **25**, 611.

Kauffmann, Beisswenger, *Ber.*, 1904, **37**, 2610.

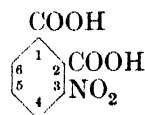
**4-Nitrophthalanil** (4-Nitrophthalic acid anil, 4-nitro-N-phenylphthalimide).

Yellow needles from EtOH- $Me_2CO$ . M.p.  $194^\circ$  ( $192^\circ$ ). Sol.  $Me_2CO$ . Spar. sol. EtOH.

Graebe, Buenzod, *Ber.*, 1899, **32**, 1993.

Bogert, Boroschek, *J. Am. Chem. Soc.*, 1901, **23**, 756.

**3-Nitrophthalic Acid**



$C_8H_5O_6N$

MW, 211

Pale yellow prisms from  $H_2O$ . M.p.  $218^\circ$ . Mod. sol. hot  $H_2O$ . Sol. hot MeOH, EtOH. Spar. sol.  $Et_2O$ . Insol.  $CHCl_3$ ,  $CCl_4$ ,  $CS_2$ ,  $C_6H_6$ , pet. ether.  $k$  (first) =  $1.22 \times 10^{-2}$  at  $25^\circ$ ; (second) =  $4 \times 10^{-5}$  at  $25^\circ$ . Above  $230^\circ \rightarrow$  anhydride.

1-Me ester:  $C_9H_7O_6N$ . MW, 225. Pale yellowish leaflets from hot  $H_2O$ . M.p.  $164^\circ$  ( $157^\circ$ ).  $k = 1.66 \times 10^{-2}$  at  $25^\circ$ . Conc.  $NH_3$ . Aq.  $\rightarrow$  3-nitro-1-phthalamic acid. 1-Menthyl ester: m.p.  $149-50^\circ$ .  $[\alpha]_D^{20} = -102^\circ$  in  $C_6H_6$ . Chloride:  $C_9H_6O_5NCl$ . MW, 243.5. Cryst. from pet. ether. M.p.  $97-9^\circ$ .

2-Me ester: cryst. +  $H_2O$ . M.p. anhyd.  $152-3^\circ$ .  $k = 2.1 \times 10^{-3}$  at  $25^\circ$ . 1-Menthyl ester: m.p.  $66-7^\circ$ .  $[\alpha]_D^{20} = -122.9^\circ$  in  $C_6H_6$ . Chloride: cryst. from pet. ether. M.p.  $95-7^\circ$ .

Di-Me ester:  $C_{10}H_9O_6N$ . MW, 239. Pale yellowish cryst. from MeOH. Aq. M.p.  $68-9^\circ$ .

1-Et ester:  $C_{10}H_9O_6N$ . MW, 239. Needles +  $2\frac{1}{2}H_2O$  from  $H_2O$ . M.p.  $53^\circ$ , anhyd.  $112^\circ$ . 1-Menthyl ester: m.p.  $99^\circ$ .  $[\alpha]_D^{20} = -93^\circ$  in  $C_6H_6$ . Chloride:  $C_{10}H_8O_5NCl$ . MW, 257.5. Cryst. from pet. ether. M.p.  $58-60^\circ$ .

2-Et ester: yellowish needles from EtOH, prisms from  $CHCl_3$ . M.p.  $157^\circ$ . 1-Menthyl ester: m.p.  $57-8^\circ$ .  $[\alpha]_D^{20} = -125^\circ$  in  $C_6H_6$ . Chloride: cryst. from pet. ether. M.p.  $74-6^\circ$ .

Di-Et ester:  $C_{12}H_{13}O_6N$ . MW, 267. Prisms from EtOH, needles from pet. ether. M.p.  $46^\circ$ . Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

1-Propyl ester:  $C_{11}H_{11}O_6N$ . MW, 253. M.p.  $122-3^\circ$ . 1-Menthyl ester: m.p.  $89^\circ$ .  $[\alpha]_D^{20} = -83^\circ$  in  $C_6H_6$ . Chloride:  $C_{11}H_{10}O_5NCl$ . MW, 271.5. Cryst. from pet. ether. M.p.  $118^\circ$ .

2-Propyl ester: m.p.  $141-2^\circ$  ( $138-9^\circ$ ). 1-Menthyl ester: m.p.  $38-40^\circ$ .  $[\alpha]_D^{20} = -116.9^\circ$  in  $C_6H_6$ . Chloride: cryst. from pet. ether. M.p.  $32^\circ$ .

2-Isopropyl ester: cryst. M.p.  $152-3^\circ$ .

2-Butyl ester :  $C_{12}H_{13}O_6N$ . MW, 267. Cryst. M.p. 146–7°.

1-Isobutyl ester : m.p. 149°. 1-Menthyl ester : m.p. 76–7°.  $[\alpha]_D^{20} = 79^\circ$  in  $C_6H_6$ .

2-Isobutyl ester : m.p. 176–7°. 1-Menthyl ester : liq.  $[\alpha]_D^{20} = 115.7^\circ$  in  $C_6H_6$ .

1-d-Amyl ester :  $C_{13}H_{15}O_6N$ . MW, 281. Cryst. from  $C_6H_6$ . M.p. 116°.

2-d-Amyl ester : leaflets from  $C_6H_6$ . M.p. 157.5–158.5°. Very sol.  $Me_2CO$ . Sol. EtOH,  $CHCl_3$ . Spar. sol.  $CS_2$ ,  $C_6H_6$ .  $[\alpha]_D^{21} + 2.2^\circ$  in  $Me_2CO$ ,  $+ 2.7^\circ$  in EtOH.

2-l-Amyl ester :  $[\alpha]_D^{20} = 2.7^\circ$  in EtOH. Brucine salt : cryst. from EtOH.Aq. M.p. 105°.

1-dl-Amyl ester : yellowish prisms from  $C_6H_6$ . M.p. 117°.

2-dl-Amyl ester : cryst. from  $C_6H_6$ . M.p. 156–8°.

1-Isoamyl ester : exists in two forms. Stable form, m.p. 95° (93.5°); labile form, m.p. 78°. Very sol. EtOH,  $Me_2CO$ . Sol.  $C_6H_6$ , hot  $CCl_4$ . Spar. sol.  $CS_2$ , ligroin.

2-Isoamyl ester : cryst. from  $C_6H_6$ . M.p. 165–6°. Spar. sol.  $C_6H_6$ ,  $CCl_4$ .

2-l-Menthyl ester :  $C_{18}H_{23}O_6N$ . MW, 349. M.p. 160–2°.

1-Benzyl ester :  $C_{15}H_{11}O_6N$ . MW, 301. M.p. 165°.

2-Benzyl ester : m.p. 174°. 1-Menthyl ester :  $[\alpha]_D^{20} = 83^\circ$  in  $C_6H_6$ . Chloride :  $C_{15}H_{10}O_6NCl$ . MW, 319.5. Cryst. from pet. ether. M.p. 74–5°.

Dichloride :  $C_6H_3O_4NCl_2$ . MW, 248. Prisms from  $Et_2O$ . M.p. 76–7°. Spar. sol.  $CCl_4$ , ligroin. Dry  $NH_3 \rightarrow$  3-nitrophthalimide.  $NH_3.Aq. \rightarrow$  3-nitrophthalamic acid.

1-Amide : see 3-Nitrophthalamic Acid.

Diamide :  $C_8H_7O_4N_3$ . MW, 209. Cryst. powder. M.p. 200–1° decomp. Above m.p.  $\rightarrow$  3-nitrophthalimide.

Imide : see 3-Nitrophthalimide.

Anil : see 3-Nitrophthalanil.

Anilide :  $C_{14}H_{10}O_6N_2$ . MW, 286. Pale yellow needles from  $AcOEt$ . M.p. 181°. Sol. EtOH. Spar. sol. cold  $H_2O$ . Reacts acid to litmus. Above m.p.  $\rightarrow$  3-nitrophthalanil.

Dianilide :  $C_{20}H_{15}O_4N_3$ . MW, 361. Needles from EtOH. M.p. 233–4°. Sol. hot EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ . Spar. sol.  $C_6H_6$ , toluene.

Di-m-nitroanilide : needles from EtOH. M.p. 225–30° decomp.

Di-p-nitroanilide : m.p. 197–200°.

Di-p-toluidide : m.p. 223–4° decomp.

1-Nitrile : 6-nitro-2-cyanobenzoic acid.  $C_8H_4O_4N_2$ . MW, 192. Needles. M.p. 99–100°, resolidifies and then has m.p. 214–15°.

1-Hydrazide : needles. Does not melt below 280°.  $B,N_2H_4$  : needles from EtOH.Aq. M.p. 157°. N-Benzylidene : needles from EtOH. M.p. 177°.

Anhydride : see 3-Nitrophthalic Anhydride.

Cohen, Woodroffe, Anderson, *J. Chem. Soc.*, 1916, 109, 222.

Cohen, Marshall, Woodman, *J. Chem. Soc.*, 1915, 107, 893.

Wegscheider, Lipschitz, *Monatsh.*, 1900, 21, 787.

Kahn, *Ber.*, 1902, 35, 3861.

Miller, *Ann.*, 1881, 208, 243.

Bogert, Boroschek, *J. Am. Chem. Soc.*, 1901, 23, 745.

McKenzie, *J. Chem. Soc.*, 1901, 79, 1136.

Marckwald, *Ber.*, 1902, 35, 1604.

Marckwald, McKenzie, *Ber.*, 1901, 34, 486.

Chambers, *J. Am. Chem. Soc.*, 1903, 25, 607.

Tingle, Bates, *J. Am. Chem. Soc.*, 1910, 32, 1325.

Culhane, Woodward, *Organic Syntheses*, Collective Vol. I, 399.

#### 4-Nitrophthalic Acid.

Pale yellowish needles from  $Et_2O$ . M.p. 165° (160–1°). Sol.  $H_2O$ , EtOH, hot  $AcOEt$ . Insol.  $CHCl_3$ ,  $CCl_4$ ,  $CS_2$ ,  $C_6H_6$ , pet. ether.  $k$  (first) =  $7.6 \times 10^{-3}$  at 25°; (second) =  $4 \times 10^{-5}$  at 25°. Above m.p.  $\rightarrow$  anhydride.

1-Me ester : cryst. +  $H_2O$  from  $H_2O$ . M.p. anhyd. 131–2°.  $k = 4.6 \times 10^{-3}$  at 25°.

1-Menthyl ester : liq.  $[\alpha]_D^{20} = 61.6^\circ$  in  $C_6H_6$ .

2-Me ester : yellow needles from  $C_6H_6$ . M.p. 140–2°.

Di-Me ester : cryst. from EtOH.Aq. M.p. 65–6°.

1-Et ester : needles from  $H_2O$ . M.p. 127–8°. Sol.  $H_2O$ ,  $Et_2O$ ,  $C_6H_6$ .  $k = 3.05 \times 10^{-3}$  at 25°.

2-Et ester : needles from  $C_6H_6$ -pet. ether. M.p. 137°. Sol.  $H_2O$ .  $k = 5.2 \times 10^{-3}$  at 25°.

Di-Et ester : plates from EtOH. M.p. 33–4°. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

Diamide : cryst. M.p. 200° decomp. Above m.p.  $\rightarrow$  4-nitrophthalimide.

Imide : see 4-Nitrophthalimide.

Anil : see 4-Nitrophthalanil.

Anilide : pale yellow cryst. from  $AcOEt$ . M.p. 192°. Sol. EtOH. Insol.  $H_2O$ ,  $Et_2O$ . Reacts acid to litmus. Above m.p.  $\rightarrow$  4-nitrophthalanil.

p-Toluidide : needles. M.p. 172°. Insol. cold  $H_2O$ ,  $Et_2O$ .

$\beta$ -Naphthylamide : pale yellow cryst. from

$\text{Me}_2\text{CO}-\text{C}_6\text{H}_5$ . M.p.  $202-4^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ .  $\text{Me}_2\text{CO}$ , AcOEt. Spar. sol.  $\text{CHCl}_3$ . Insol.  $\text{C}_6\text{H}_6$ .  
*Anhydride*: see 4-Nitrophthalic Anhydride.

Bogert, Boroschek, *J. Am. Chem. Soc.*, 1901, **23**, 752.

Miller, *Ann.*, 1881, **208**, 225.

Seidel, Bittner, *Monatsh.*, 1902, **23**, 418.

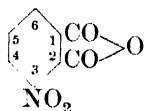
Wegscheider, Lipschitz, *Monatsh.*, 1900, **21**, 804.

Wegscheider, Bondi, *Monatsh.*, 1905, **26**, 1048.

Wegscheider, Kušý, Dúbrav, *Monatsh.*, 1903, **24**, 825.

Tingle, Bates, *J. Am. Chem. Soc.*, 1910, **32**, 1328.

### 3-Nitrophthalic Anhydride



$\text{C}_8\text{H}_5\text{O}_5\text{N}$

MW, 193

Needles from AcOH or  $\text{Me}_2\text{CO}$ . M.p.  $164^\circ$ . Sol. hot AcOH. Mod. sol.  $\text{Me}_2\text{CO}$ , hot EtOH. Spar. sol.  $\text{C}_6\text{H}_6$ .

Lawrance, *J. Am. Chem. Soc.*, 1920, **42**, 1871.

Nicolet, Bender, *Organic Syntheses*. Collective Vol. I, 402.

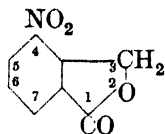
### 4-Nitrophthalic Anhydride.

Needles from  $\text{CHCl}_3$ -pet. ether. M.p.  $119^\circ$  ( $114^\circ$ ). Sol. usual org. solvents. Spar. sol.  $\text{Et}_2\text{O}$ . Insol. pet. ether.

See first reference above and also

Crossley, Renouf, *J. Chem. Soc.*, 1909, **95**, 208.

### 4-Nitrophthalide



$\text{C}_8\text{H}_5\text{O}_4\text{N}$

MW, 179

Leaflets from MeOH. M.p.  $136^\circ$ . Sol. EtOH,  $\text{CHCl}_3$ , AcOH,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ .  $\text{CrO}_3 + \text{AcOH} \rightarrow$  3-nitrophthalic acid.

Beilstein, Kurbatow, *Ann.*, 1880, **202**, 219.

Borsche, Diacont, Hanau, *Ber.*, 1934, **67**, 676.

### 6-Nitrophthalide.

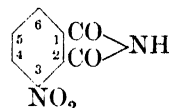
Yellowish needles from AcOH. M.p.  $145^\circ$  ( $141^\circ$ ). Very sol. hot  $\text{CHCl}_3$ , AcOH,  $\text{C}_6\text{H}_6$ . Mod. sol. EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ . Dil.  $\text{HNO}_3$  at  $140^\circ \rightarrow$  4-nitrophthalic acid.  $\text{Sn} + \text{HCl} \rightarrow$  6-aminophthalide.

See last reference above and also

Hoenig, *Ber.*, 1885, **18**, 3447.

Tasman, *Rec. trav. chim.*, 1927, **46**, 653.

### 3-Nitrophthalimide



$\text{C}_8\text{H}_4\text{O}_4\text{N}_2$

MW, 192

Pale yellow leaflets from EtOH or needles from  $\text{Me}_2\text{CO}$ . M.p.  $217-18^\circ$ . Sol. hot EtOH,  $\text{Me}_2\text{CO}$ , AcOH. Spar. sol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ , ligroin. Dil. sols. in EtOH and AcOH show blue fluor. Sublimes easily.

N-Me:  $\text{C}_9\text{H}_5\text{O}_4\text{N}_2$ . MW, 206. Needles from  $\text{CCl}_4$ . M.p.  $112-13^\circ$ .

N-Et:  $\text{C}_{10}\text{H}_8\text{O}_4\text{N}_2$ . MW, 220. Yellowish needles from EtOH, plates from  $\text{CCl}_4$ . M.p.  $105-6^\circ$ . Sol. hot EtOH. Spar. sol. hot  $\text{H}_2\text{O}$ .

N-β-Bromoethyl: plates from  $\text{CCl}_4$ . M.p.  $115-16^\circ$ .

N-Propyl:  $\text{C}_{11}\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 234. Needles from  $\text{CCl}_4$ . M.p.  $84-5^\circ$ .

N-Butyl:  $\text{C}_{12}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 248. Prisms from  $\text{CCl}_4$ . M.p.  $71-2^\circ$ .

N-Amyl:  $\text{C}_{13}\text{H}_{14}\text{O}_4\text{N}_2$ . MW, 262. Plates from  $\text{CCl}_4$ . M.p.  $93-4^\circ$ .

N-Allyl:  $\text{C}_{11}\text{H}_8\text{O}_4\text{N}_2$ . MW, 232. Plates from  $\text{CCl}_4$ . M.p.  $100-1^\circ$ .

N-Phenyl: see 3-Nitrophthalanil.

N-o-Nitrophenyl: yellow cryst. from EtOH- $\text{Me}_2\text{CO}$ . M.p.  $167^\circ$ .

N-m-Nitrophenyl: light brown cryst. from EtOH- $\text{Me}_2\text{CO}$ . M.p.  $219^\circ$ .

N-p-Nitrophenyl: yellow cryst. from  $\text{Me}_2\text{CO}$ . M.p.  $249^\circ$ . Mod. sol.  $\text{Me}_2\text{CO}$ .

N-o-Tolyl: pale yellow needles from EtOH- $\text{Me}_2\text{CO}$ . M.p.  $145^\circ$ . Sol.  $\text{Me}_2\text{CO}$ . Spar. sol. EtOH.

N-m-Tolyl: yellow needles from EtOH- $\text{Me}_2\text{CO}$ . M.p.  $129^\circ$ .

N-p-Tolyl: pale yellow needles from EtOH- $\text{Me}_2\text{CO}$ . M.p.  $154^\circ$ .

N-Benzyl:  $\text{C}_{15}\text{H}_{10}\text{O}_4\text{N}_2$ . MW, 282. Needles from  $\text{CCl}_4$ . M.p.  $142-3^\circ$ .

**N-p-Nitrobenzyl**: prisms from AcOH. M.p. 181–2°.

Bogert, Boroschek, *J. Am. Chem. Soc.*, 1901, **23**, 747.

Bogert, Chambers, *J. Am. Chem. Soc.*, 1905, **27**, 652.

Kahn, *Ber.*, 1902, **35**, 3862.

Seidel, Bittner, *Monatsh.*, 1902, **23**, 420.

Chapman, Stephen, *J. Chem. Soc.*, 1925, 1795.

Sah, Ma, *Ber.*, 1932, **65**, 1630.

#### 4-Nitrophthalimide.

Needles from H<sub>2</sub>O, yellowish leaflets from EtOH–Me<sub>2</sub>CO. M.p. 202° (193–5°). Sol. EtOH, AcOH, Me<sub>2</sub>CO. Spar. sol. hot H<sub>2</sub>O.

**N-Me**: needles from EtOH. M.p. 179–80°. Sol. Me<sub>2</sub>CO, AcOEt, CHCl<sub>3</sub>, hot EtOH. Mod. sol. C<sub>6</sub>H<sub>6</sub>, Et<sub>2</sub>O, hot H<sub>2</sub>O.

**N-Et**: pale yellow leaflets. M.p. 111–12°. Spar. sol. hot H<sub>2</sub>O.

**N-Phenyl**: see 4-Nitrophthalanil.

**N-o-Nitrophenyl**: pale yellow needles from PhNO<sub>2</sub>. M.p. 233°. Spar. sol. EtOH, Me<sub>2</sub>CO.

**N-m-Nitrophenyl**: yellow needles from PhNO<sub>2</sub>. M.p. 243°. Spar. sol. EtOH, Me<sub>2</sub>CO.

**N-p-Nitrophenyl**: yellow cryst. from PhNO<sub>2</sub>. M.p. 251–3°. Spar. sol. Me<sub>2</sub>CO. Insol. EtOH.

**N-o-Tolyl**: brownish cryst. powder. M.p. 160°. Sol. Me<sub>2</sub>CO. Spar. sol. EtOH.

**N-m-Tolyl**: light brownish cryst. from PhNO<sub>2</sub>. M.p. 197°. Spar. sol. Me<sub>2</sub>CO. Insol. EtOH.

**N-p-Tolyl**: yellow cryst. from EtOH–Me<sub>2</sub>CO. M.p. 165°. Sol. Me<sub>2</sub>CO. Spar. sol. EtOH.

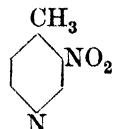
Bogert, Boroschek, *J. Am. Chem. Soc.*, 1901, **23**, 755.

Seidel, Bittner, *Monatsh.*, 1902, **23**, 420.

Chapman, Stephen, *J. Chem. Soc.*, 1925, 1796.

Levy, Stephen, *J. Chem. Soc.*, 1931, 79.

#### 3-Nitro-γ-picoline



C<sub>6</sub>H<sub>6</sub>O<sub>2</sub>N<sub>2</sub>

MW, 138

Deliquescent cryst.

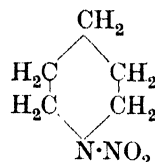
**B,HCl**: prisms from EtOH–pet. ether. M.p. 178°.

**B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>**: yellow needles. M.p. 267°.

**Picrate**: golden-yellow plates. M.p. 118°.

Koenigs, Fulde, *Ber.*, 1927, **60**, 2108.

#### N-Nitropiperidine



C<sub>5</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

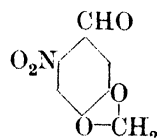
MW, 130

M.p. –5.5°. B.p. 245°/765 mm. slight decomp.  $D_4^{26.4}$  1.1519.  $n_D^{26.4}$  1.4954. Sol. hot H<sub>2</sub>O. Volatile in steam.

Bamberger, Kirpal, *Ber.*, 1895, **28**, 536.

Franchimont, van Rijn, Friedmann, *Rec. trav. chim.*, 1907, **26**, 230.

#### 6-Nitropiperonal



C<sub>8</sub>H<sub>5</sub>O<sub>5</sub>N

MW, 195

Needles from H<sub>2</sub>O or AcOEt–EtOH. M.p. 98.5° (95.5°). Sol. EtOH, boiling H<sub>2</sub>O. Sublimes. Turns yellow in light. 2% KMnO<sub>4</sub> → 6-nitropiperonylic acid.

**Oxime**: yellow needles from EtOH. M.p. 212° (203°). Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Insol. H<sub>2</sub>O, pet. ether. **Acetyl**: yellow needles from EtOH. M.p. 142°.

**Semicarbazone**: decomp. at 272–8°.

**Phenylhydrazone**: red needles from EtOH. M.p. 218.5° decomp. (212° decomp.).

**p-Nitrophenylhydrazone**: m.p. 248° decomp.

**Azine**: m.p. 257° decomp.

**Semioxamazone**: m.p. 325°.

**o-Tolylimide**: yellow needles. M.p. 128°.

**p-Tolylimide**: yellow needles from EtOH. M.p. 121.5°.

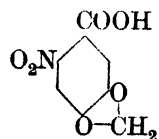
**Di-Me acetal**: needles from MeOH. M.p. 69°. Insol. ligroin.

Parijs, *Rec. trav. chim.*, 1930, **49**, 17.

Bogert, Elder, *J. Am. Chem. Soc.*, 1929, **51**, 534.

Ekeley, Klemme, *J. Am. Chem. Soc.*, 1928, **50**, 2711.

#### 6-Nitropiperonylic Acid



C<sub>8</sub>H<sub>5</sub>O<sub>6</sub>N

MW, 211

Yellow needles from  $\text{H}_2\text{O}$ . M.p.  $172^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ .

*K salt*: yellow needles  $\div \frac{1}{2}\text{H}_2\text{O}$ . Very sol.  $\text{H}_2\text{O}$ .

*Cu salt*: green cryst.  $+ 4\text{H}_2\text{O}$ .

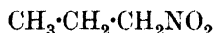
*Me ester*:  $\text{C}_5\text{H}_7\text{O}_6\text{N}$ . MW, 225. Needles from EtOH. M.p.  $102^\circ$ . Sol. warm EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ , pet. ether.

Jobst, Hesse, *Ann.*, 1879, **199**, 70.

Oertly, Pictet, *Ber.*, 1910, **43**, 1336.

Mosettig, Czadek, *Monatsh.*, 1931, **57**, 301.

### 1-Nitropropane



$\text{C}_3\text{H}_7\text{O}_2\text{N}$  MW, 89

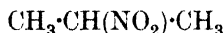
Oil. B.p.  $130.5\text{--}131.5^\circ$ .  $D_4^{20}$  1.0221,  $D_4^{25}$  1.0081.  $n_D^{25}$  1.40027. Non-miscible with  $\text{H}_2\text{O}$ . Heat of comb.  $\text{C}_6$  477.8 Cal.

Neogi, Chowdhuri, *J. Chem. Soc.*, 1916, **109**, 703.

Neogi, *J. Chem. Soc.*, 1914, **105**, 2375.

Meyer, *Ann.*, 1874, **171**, 36.

### 2-Nitropropane



$\text{C}_3\text{H}_7\text{O}_2\text{N}$  MW, 89

Liq. B.p.  $115\text{--}18^\circ$  ( $117\text{--}20^\circ$ ).  $D^0$  1.024.

See previous references.

### 1-Nitropropionic Acid



$\text{C}_3\text{H}_5\text{O}_4\text{N}$  MW, 119

Needles from  $\text{CS}_2$ . M.p.  $61\text{--}61.5^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{CS}_2$ , ligroin.

*Et ester*:  $\text{C}_5\text{H}_9\text{O}_4\text{N}$ . MW, 147. B.p.  $190.5^\circ$ ,  $174^\circ/390$  mm.,  $80.5^\circ/10.5$  mm. Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{H}_2\text{O}$ . *Na salt*: needles from EtOH. M.p.  $200^\circ$ . *NH<sub>4</sub> salt*: cryst. from EtOH. M.p.  $119^\circ$ .

*Amide*:  $\text{C}_3\text{H}_6\text{O}_3\text{N}_2$ . MW, 118. Needles from  $\text{CHCl}_3$  or  $\text{Et}_2\text{O}$ . M.p.  $68\text{--}9^\circ$ . *NH<sub>4</sub> salt*: m.p.  $127\text{--}8^\circ$ .

Steinkopf, Supan, *Ber.*, 1910, **43**, 3246.

Ulpiani, *Atti accad. Lincei*, 1903, **12**, i, 442.

Schmidt, Widman, *Ber.*, 1909, **42**, 1893.

### 2-Nitropropionic Acid



$\text{C}_3\text{H}_5\text{O}_4\text{N}$  MW, 119

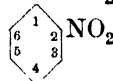
Cryst. from  $\text{CHCl}_3$ . M.p.  $66\text{--}7^\circ$ . Very sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Sol. warm  $\text{CHCl}_3$ . Insol. ligroin.  $k = 1.62 \times 10^{-4}$  at  $25^\circ$ .

*Et ester*:  $\text{C}_5\text{H}_9\text{O}_4\text{N}$ . MW, 147. B.p.  $161\text{--}5^\circ$ . *Chloride*:  $\text{C}_3\text{H}_4\text{O}_3\text{NCl}$ . MW, 137.5. B.p.  $123^\circ/10$  mm.

Lewkowitsch, *J. prakt. Chem.*, 1879, **20**, 169.

Barger, Tutin, *Biochem. J.*, 1918, **12**, 405.

**o-Nitropropiophenone** (*Ethyl 2-nitrophenyl ketone*)



$\text{C}_9\text{H}_9\text{O}_3\text{N}$  MW, 179

Cryst. from EtOH. M.p.  $85^\circ$ . B.p.  $175^\circ/25$  mm.,  $166\text{--}7^\circ/15$  mm. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

Auwers, Duesberg, *Ber.*, 1920, **53**, 1208.

Comanducci, Pescitelli, *Gazz. chim. ital.*, 1906, **36**, 790.

**m-Nitropropiophenone** (*Ethyl 3-nitrophenyl ketone*).

Yellow needles from EtOH. M.p.  $100^\circ$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Sol. boiling  $\text{H}_2\text{O}$ . Spar. sol. petrol. Insol. cold  $\text{H}_2\text{O}$ .

Comanducci, Pescitelli, *Gazz. chim. ital.*, 1906, **36**, 789.

**p-Nitropropiophenone** (*Ethyl 4-nitrophenyl ketone*).

Yellow cryst. M.p.  $114^\circ$ . Sol. hot  $\text{H}_2\text{O}$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

Comanducci, Pescitelli, *Gazz. chim. ital.*, 1906, **36**, 790.

### 2-Nitropropyl Alcohol



$\text{C}_3\text{H}_7\text{O}_3\text{N}$  MW, 105

Thick liq. B.p.  $120\text{--}2^\circ/32$  mm.  $D^0$  1.209. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ .

Henry, *Chem. Zentr.*, 1897, I, 741.

### 3-Nitropropyl Alcohol



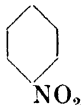
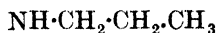
$\text{C}_3\text{H}_7\text{O}_3\text{N}$  MW, 105

Thick oil. B.p.  $138\text{--}40^\circ/32$  mm.  $D^{18}$  1.173. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ .

Henry, *Chem. Zentr.*, 1897, II, 337.

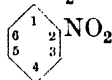
### N-Nitropropylamine.

See Propylnitramine.

4-Nitro-*N*-propylanilineC<sub>9</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>

MW, 180

Yellow cryst. from EtOH. M.p. 64–5°.

Jaeger, v. Kregten, *Chem. Zentr.*, 1912, I, 1302.*o*-Nitropropylbenzene (*o*-Nitrophenylpropane)C<sub>9</sub>H<sub>11</sub>O<sub>2</sub>N

MW, 165

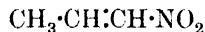
Yellow oil. B.p. 133°/26 mm. Odour similar to nitrobenzene. Volatile in steam.

Brady, Cunningham, *J. Chem. Soc.*, 1934, 122.*m*-Nitropropylbenzene (*m*-Nitrophenylpropane).

Yellow oil. B.p. 136°/16 mm. Volatile in steam.

See previous reference.

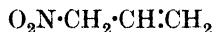
## 1-Nitropropylene

C<sub>3</sub>H<sub>5</sub>O<sub>2</sub>N

MW, 87

Liq. B.p. 37°/10 mm. D<sub>4</sub><sup>20</sup> 1.0661. n<sub>D</sub><sup>20</sup> 1.4527.Schmidt, Rutz, *Ber.*, 1928, 61, 2147.

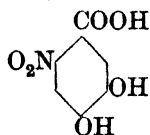
## 3-Nitropropylene (Nitroallyl)

C<sub>3</sub>H<sub>5</sub>O<sub>2</sub>N

MW, 87

Liq. B.p. 125–30°/760 mm., 87–9°/180 mm. D<sub>21</sub> 1.051. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Volatile in ether vapour. Bitter taste.Askenasy, Meyer, *Ber.*, 1892, 25, 1701.

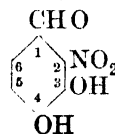
## 6-Nitroprotocatechuic Acid (6-Nitro-3 : 4-dihydroxybenzoic acid)

C<sub>7</sub>H<sub>5</sub>O<sub>6</sub>N

MW, 199

4-*Me* ether : see 6-Nitroisovanillic Acid.Di-*Me* ether : see 6-Nitroveratric Acid.Di-Et ether : C<sub>11</sub>H<sub>13</sub>O<sub>6</sub>N. MW, 255. Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 145–8°.Perkin, Watson, *J. Chem. Soc.*, 1915, 107, 206.

## 2-Nitroprotocatechuic Aldehyde (2-Nitro-3 : 4-dihydroxybenzaldehyde)

C<sub>7</sub>H<sub>5</sub>O<sub>5</sub>N

MW, 183

Yellow needles from Et<sub>2</sub>O-ligroin. M.p. 176°.3-*Me* ether : see 2-Nitrovanillin.4-*Me* ether : see 2-Nitroisovanillin.Di-*Me* ether : see 2-Nitroveratric Aldehyde.Hayduck, *Ber.*, 1903, 36, 3528.

## 5-Nitroprotocatechuic Aldehyde (5-Nitro-3 : 4-dihydroxybenzaldehyde).

Yellow needles from toluene or H<sub>2</sub>O. M.p. 106°. Very sol. H<sub>2</sub>O with yellowish-red col.3-*Me* ether : see 5-Nitrovanillin.4-*Me* ether : see 5-Nitroisovanillin.Di-*Me* ether : see 5-Nitroveratric Aldehyde.Hayduck, *Ber.*, 1903, 36, 2933.

## 6-Nitroprotocatechuic Aldehyde (6-Nitro-3 : 4-dihydroxybenzaldehyde).

Pale yellow cryst. from 20% EtOH. M.p. 203° decomp. FeCl<sub>3</sub> → dark green col. NaOH → reddish-violet sol.

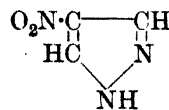
Azine : orange-yellow amorph. ppt. M.p. about 241°.

Semicarbazone : orange ppt. M.p. 254° decomp.

Phenylhydrazone : reddish-brown ppt. M.p. 203° decomp.

*p*-Nitrophenylhydrazone : red ppt. M.p. 290° decomp.3-*Me* ether : see 6-Nitrovanillin.4-*Me* ether : see 6-Nitroisovanillin.Di-*Me* ether : see 6-Nitroveratric Aldehyde.Parijs, *Rec. trav. chim.*, 1930, 49, 33.

## 4-Nitropyrazole

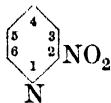
C<sub>3</sub>H<sub>3</sub>O<sub>2</sub>N<sub>3</sub>

MW, 113

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 162°. B.p. 323°.Buchner, Fritsch, *Ann.*, 1893, 273, 265.Knorr, *Ann.*, 1894, 279, 228.Hill, Torrey, *Am. Chem. J.*, 1899, 22, 105.



## 2-Nitropyridine



$C_5H_4O_2N_2$  MW, 124  
Yellow cryst. M.p. 71°. B.p. 256°. Volatile in steam.

Hertog, Overhoff, *Rec. trav. chim.*, 1930, **49**, 552.

Kirpal, Böhm, *Ber.*, 1931, **64**, 767; 1932, **65**, 680.

## 3-Nitropyridine.

Needles from  $C_6H_6$ -ligroin. M.p. 41°. B.p. 216°. Volatile in steam.

$B.HCl$ : leaflets. M.p. 154°.

$B.HNO_3$ : cryst. from dil.  $HNO_3$ . M.p. 150–1°.

$B_2, AgNO_3$ : needles. M.p. 175–6°.

$B.HAuCl_4$ : yellow needles. M.p. 140°.

$B_2, H_2PtCl_6$ : yellow needles. Decomp. at 254°.

Hertog, Overhoff, *Rec. trav. chim.*, 1930, **49**, 552.

Freytag, *J. prakt. Chem.*, 1933, **139**, 44.

Kirpal, Reiter, *Ber.*, 1925, **58**, 699.

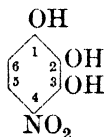
Räth, Swiss P., 127,257, (*Chem. Abstracts*, 1929, **23**, 1143).

## 4-Nitropyridine.

Plates from EtOH.Aq. M.p. 50°.

Kirpal, Böhm, *Ber.*, 1932, **65**, 680.

## 4-Nitropyrogallol (4-Nitro-1 : 2 : 3-tri-hydroxybenzene)



$C_6H_5O_5N$  MW, 171

Yellow needles from  $H_2O$  or  $C_6H_6$ . M.p. 162°. Sol. EtOH, Et<sub>2</sub>O, AcOH, hot  $H_2O$ , hot  $C_6H_6$ .  $FeCl_3$  → green col.

1 : 3-Di-Me ether:  $C_8H_9O_5N$ . MW, 199. Yellow needles +  $H_2O$  from  $H_2O$ . M.p. 67–8°. Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ , AcOH. Acetyl: yellow cryst. from EtOH. M.p. 92–3°. Sol.  $Me_2CO$ ,  $CHCl_3$ . Less sol. AcOH. Insol.  $H_2O$ .

Tri-Me ether:  $C_9H_{11}O_5N$ . MW, 213. Yellow cryst. from EtOH. M.p. 44°. Sol. Et<sub>2</sub>O. Mod. sol. EtOH. Insol.  $H_2O$ .

Triacetyl: yellow needles from  $C_6H_6$ -pet. ether. M.p. 85°. Sol. Et<sub>2</sub>O. Spar. sol. EtOH,  $C_6H_6$ . Insol. pet. ether.

Benzoyl deriv.: needles from  $C_6H_6$ . M.p. 214° decomp. Sol. Et<sub>2</sub>O. Spar. sol.  $H_2O$ , Et<sub>2</sub>O. Alc.  $FeCl_3$  → weak green col.

1-(or 3)-Carboethoxyl: yellow leaflets from EtOH.Aq. M.p. 134°.  $FeCl_3$  → green col.

Einhorn, Cobliner, Pfeiffer, *Ber.*, 1904, **37**, 114.

Brand, Collischonn, *J. prakt. Chem.*, 1922, **103**, 329.

## 5-Nitropyrogallol (5-Nitro-1 : 2 : 3-tri-hydroxybenzene).

Brownish-yellow prisms or needles +  $H_2O$  from  $H_2O$ . M.p. 205° decomp. Spar. sol. cold  $H_2O$ . Lime water → dark red col.  $FeCl_3$  → green col.

1 : 2-(or 1 : 3)-Di-Me ether: pale yellow cryst. from EtOH.Aq. M.p. 112–14°. Sol. KOH with orange col.

Tri-Me ether: yellow needles from EtOH, prisms from AcOH. M.p. 100° (99°). Sol. hot EtOH.

Tri-Et ether:  $C_{12}H_{17}O_5N$ . MW, 255. Needles from EtOH. M.p. 74°.

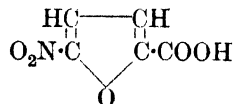
Harding, *J. Chem. Soc.*, 1914, **105**, 2797.

Schiffer, *Ber.*, 1892, **25**, 722.

Pollak, Goldstein, *Ann.*, 1907, **352**, 168.

Barth, *Monatsh.*, 1880, **1**, 882.

See also Einhorn, Cobliner, Pfeiffer, *Ber.*, 1904, **37**, 104.

5-Nitropyromucic Acid (5-Nitro- $\alpha$ -furoic acid, 5-nitrofuran-2-carboxylic acid)

$C_5H_3O_5N$  MW, 157

Pale yellow plates from  $H_2O$ . M.p. 184°. Sol. boiling  $H_2O$ , EtOH, Et<sub>2</sub>O. Spar. sol.  $C_6H_6$ . Insol.  $CHCl_3$ . Sublimes.

Me ester:  $C_6H_5O_5N$ . MW, 171. Yellowish leaflets. M.p. 78–5°.

Et ester:  $C_7H_7O_5N$ . MW, 185. Leaflets. M.p. 101°. Sol. boiling EtOH. Spar. sol. Et<sub>2</sub>O,  $C_6H_6$ .

Chloride:  $C_5H_3O_4NCl$ . MW, 175.5. Leaflets from  $CHCl_3$ . M.p. 38°. Sol.  $CHCl_3$ , Et<sub>2</sub>O. Insol. pet. ether.

Amide:  $C_5H_4O_4N_2$ . MW, 156. Cryst. from EtOH. M.p. 161°. Mod. sol. EtOH. Spar. sol.  $H_2O$ , Et<sub>2</sub>O.

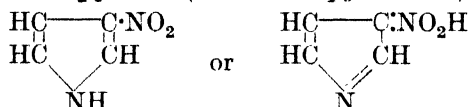
Anilide:  $C_{11}H_8O_4N_2$ . MW, 232. Yellow needles from EtOH. M.p. 180°. Spar. sol. EtOH. Insol.  $H_2O$ , Et<sub>2</sub>O.

*p*-Toluidide:  $C_{12}H_{10}O_4N_2$ . MW, 246. Yellow prisms. M.p. 162°.

Marquis, *Ann. chim. phys.*, 1905, **4**, 256.

Rinkes, *Rec. trav. chim.*, 1930, **49**, 1169.

### 3-Nitropyrrole (3-Isonitrosopyrrolenine)



$C_4H_4O_2N_2$  MW, 112

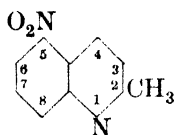
Yellowish prisms or leaflets from ligroin. M.p. 63-5°. At 70° → a dimer.

*Dimer*:  $C_8H_8O_4N_4$ . MW, 224. Yellowish prisms from  $H_2O$ . M.p. 101°. Sol. EtOH,  $Me_2CO$ , AcOH, AcOEt. Spar. sol.  $H_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $CCl_4$ , ligroin.

Hale, Hoyt, *J. Am. Chem. Soc.*, 1915, **37**, 2550.

Angeli, Alessandri, *Atti accad. Lincei*, 1911, **20**, i, 311.

### 5-Nitroquinaldine (5-Nitro-2-methylquinoline)



$C_{10}H_8O_2N_2$  MW, 188

Needles from EtOH.Aq. M.p. 82°. Sol.  $Et_2O$ . Spar. sol.  $H_2O$ . Volatile in steam.  $C_6H_5 \cdot CHO$  at 150° → 5-nitro-2-styrylquinoline.

*Methiodide*: orange cryst. from EtOH. M.p. 201°.

*Methopicate*: yellow needles. M.p. 151-5°.

Decker, Remfry, *Ber.*, 1905, **38**, 2775.

Doebner, Miller, *Ber.*, 1884, **17**, 1700.

### 6-Nitroquinaldine.

Yellow needles from  $H_2O$ . M.p. 173-4° (164°). Sol. EtOH. Spar. sol.  $H_2O$ . Insol.  $Et_2O$ .  $C_6H_5 \cdot CHO + ZnCl_2 \rightarrow$  6-nitro-2-styrylquinoline.

*Methiodide*: green cryst. M.p. 214° decomp.

Cohn, Springer, *Monatsh.*, 1903, **24**, 99.

Hamer, *J. Chem. Soc.*, 1921, **119**, 1435.

### 8-Nitroquinaldine.

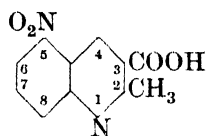
Pale yellow needles from EtOH.Aq. M.p. 137°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. cold  $H_2O$ .  $C_6H_5 \cdot CHO + ZnCl_2 \rightarrow$  8-nitro-2-styrylquinoline.

Doebner, Miller, *Ber.*, 1884, **17**, 1700.

Claus, Momberger, *J. prakt. Chem.*, 1897, **56**, 378.

Dict. of Org. Comp.—III.

### 5-Nitroquinaldine-3-carboxylic Acid



$C_{11}H_8O_4N_2$

MW, 232

Yellow needles from EtOH. M.p. 236°. Insol.  $H_2O$ . 10%  $H_2SO_4$  at 150° → 5-nitroquinaldine.

*B.HCl*: yellowish-red cryst. M.p. 215° decomp. Sol.  $H_2O$ .

*Et ester*:  $C_{13}H_{12}O_4N_2$ . MW, 260. Yellowish needles. M.p. 126°. Sol. EtOH,  $C_6H_6$ ,  $Et_2O$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ .  $B_2H_2PtCl_6$ : needles from dil. HCl. Decomp. at 232°.

Claus, Momberger, *J. prakt. Chem.*, 1897, **56**, 375.

Decker, Remfry, *Ber.*, 1905, **38**, 2775.

### 8-Nitroquinaldine-3-carboxylic Acid.

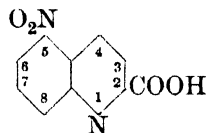
Yellowish leaflets from EtOH. M.p. 196° decomp. 10%  $H_2SO_4$  at 150° → 8-nitroquinaldine.

*B.HCl*: yellow needles. Decomp. at 204°. Spar. sol.  $H_2O$ .

*Et ester*: yellowish cryst. M.p. 137°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. hot  $H_2O$ , pet. ether. Insol. cold  $H_2O$ .  $B_2H_2PtCl_6$ : orange-yellow cryst. Decomp. at 175-95°.

See previous references.

### 5-Nitroquinaldinic Acid (5-Nitroquinoline-2-carboxylic acid)



$C_{10}H_6O_4N_2$

MW, 218

Cryst. from  $H_2O$ . M.p. 203° decomp. Above m.p. → 5-nitroquinoline.

*Ba salt*: needles from  $H_2O$ . Spar. sol.  $H_2O$ .

Besthorn, Ibele, *Ber.*, 1906, **39**, 2333.

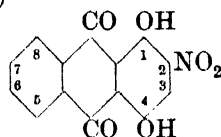
### 8-Nitroquinaldinic Acid (8-Nitroquinoline-2-carboxylic acid).

Cryst. from  $H_2O$ . M.p. 177° decomp. Above m.p. → 8-nitroquinoline.

*Ba salt*: needles from  $H_2O$ . Readily sol.  $H_2O$ .

See previous reference.

**2-Nitroquinizarin** (2-Nitro-1:4-dihydroxy-anthraquinone)



$C_{14}H_7O_6N$  MW, 285

Red needles from  $PhNO_2$ -AcOH. Dil. NaOH  $\rightarrow$  bluish-green sol. which decomposes on warming. Conc.  $H_2SO_4 \rightarrow$  red sol.  $\rightarrow$  bluish-red with boric acid.

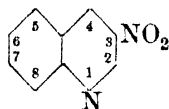
Bayer, D.R.P., 272,299, (*Chem. Zentr.*, 1914, I, 1388).

**5-Nitroquinizarin** (5-Nitro-1:4-dihydroxy-anthraquinone).

Red cryst. from AcOH. M.p.  $244-5^\circ$ . NaOH  $\rightarrow$  blue sol. Conc.  $H_2SO_4 \rightarrow$  rose-pink sol.  $\rightarrow$  yellowish-red fluor. with boric acid.

Bayer, D.R.P., 90,041.

### 3-Nitroquinoline



$C_9H_6O_2N_2$  MW, 174

Cryst. M.p.  $128^\circ$ . Volatile in steam.

Badische, D.R.P., 335,197, (*Chem. Zentr.*, 1921, II, 962).

### 5-Nitroquinoline.

Needles +  $H_2O$  from EtOH or  $Et_2O$ . M.p. anhyd.  $72^\circ$ . Sol. hot EtOH. Spar. sol. boiling  $H_2O$ . Sublimes.

$B, HCl$ : plates. M.p.  $214^\circ$  decomp.

$B, HNO_3$ : needles from  $H_2O$  or EtOH. M.p.  $191^\circ$ .

*Methiodide*: red prisms from  $H_2O$ . M.p.  $215^\circ$  decomp.

Noelting, Trautmann, *Ber.*, 1890, 23, 3655.

Knueppel, *Ber.*, 1896, 29, 706.

Decker, *J. prakt. Chem.*, 1901, 63, 573; *Ber.*, 1905, 38, 1154.

Le Fèvre, Le Fèvre, *J. Chem. Soc.*, 1935, 1472.

### 6-Nitroquinoline.

Needles from  $H_2O$  or EtOH.Aq. M.p.  $153-4^\circ$  ( $149-50^\circ$ ). Very sol.  $C_6H_6$ . Sol. EtOH, hot  $H_2O$ . Spar. sol.  $Et_2O$ , ligroin.

$B, HBr$ : cryst. M.p. about  $245^\circ$ . Insol.  $CHCl_3$ .

*Methiodide*: orange cryst. M.p. about  $245^\circ$ . Sol.  $H_2O$ . Less sol. EtOH.

*Tetrachloriodide*: yellow plates from AcOH-HCl. M.p.  $131^\circ$  decomp.

*Styphnate*: m.p.  $189.5-190.5^\circ$ .

See last reference above and also

Knueppel, *Ber.*, 1896, 29, 705.

Strache, *Monatsh.*, 1889, 10, 645.

I.G., F.P., 727,528, (*Chem. Abstracts*, 1932, 26, 5104).

Hamer, *J. Chem. Soc.*, 1921, 119, 1432.

### 7-Nitroquinoline.

Leaflets or needles from EtOH or  $H_2O$ . M.p.  $132-3^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ , hot EtOH. Sublimes.

$B, HCl$ : yellowish needles. M.p.  $225^\circ$  decomp. Hyd. by  $H_2O$ .

*Methochloride*: yellow cryst. M.p.  $212-13^\circ$  decomp.

*Methiodide*: red plates from  $H_2O$ . M.p.  $231-2^\circ$  decomp.

*Ethiodide*: red needles from EtOH. M.p.  $220^\circ$  decomp.

Knueppel, *Ber.*, 1896, 29, 706.

Claus, Massau, *J. prakt. Chem.*, 1893, 48, 170.

Bacharach, Haut, Caroline, *Rec. trav. chim.*, 1933, 52, 416.

### 8-Nitroquinoline.

Cryst. from EtOH. M.p.  $91-2^\circ$  ( $88-9^\circ$ ). Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. cold  $H_2O$ .  $KMnO_4 \rightarrow$  quinolinic acid.

Knueppel, *Ber.*, 1896, 29, 705.

Claus, Küttner, *Ber.*, 1886, 19, 2886.

Besthorn, Ibele, *Ber.*, 1906, 39, 2333.

Le Fèvre, Le Fèvre, *J. Chem. Soc.*, 1935, 1472.

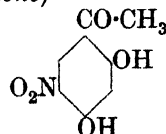
### Nitroquinoline-2-carboxylic Acid.

See Nitroquinaldinic Acid.

### Nitroquinoline-4-carboxylic Acid.

See Nitrocinchoninic Acid.

**5-Nitroresacetophenone** (5-Nitro-2:4-dihydroxyacetophenone)



$C_8H_7O_5N$  MW, 197

Yellowish needles from 50% EtOH. M.p.  $142^\circ$ .

4-Me ether: nitropeonol.  $C_9H_9O_5N$ . MW, 211. Needles from EtOH. M.p.  $155^\circ$ .

Oxime: needles from AcOH. M.p.  $238^\circ$ .

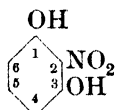
*Phenylhydrazone*: orange needles from AcOH. M.p. 215.5–216.5°.

*Di-Me ether*:  $C_{10}H_{11}O_5N$ . MW. 225. Needles from EtOH. M.p. 131°.

Nencki, Sieber, *J. prakt. Chem.*, 1881, **23**, 150.

Adams, *J. Am. Chem. Soc.*, 1919, **41**, 263.  
Lindemann, Könitzer, Romanoff, *Ann.*, 1927, **456**, 307.

## 2-Nitroresorcinol



$C_6H_5O_4N$

MW, 155

Orange-red prisms from EtOH.Aq. M.p. 85°. Volatile in steam.  $k = 0.13 \times 10^{-4}$  at 25°. Can be distilled.

*Di-Me ether*:  $C_8H_9O_4N$ . MW, 183. Yellowish needles from EtOH or AcOH. M.p. 131°. Sol.  $CHCl_3$ ,  $C_6H_6$ , hot EtOH, AcOH. Spar. sol.  $Et_2O$ . Prac. insol. ligroin. Turns greenish in sunlight. Sol. conc.  $H_2SO_4$  with red col.

*Di-Et ether*:  $C_{10}H_{13}O_4N$ . MW, 211. M.p. 106–7°.

Kauffmann, de Pay, *Ber.*, 1904, **37**, 726; D.R.P., 145,190, (*Chem. Zentr.*, 1903, II, 973).

Weselsky, Benedikt, *Monatsh.*, 1880, **1**, 887, 894.

## 4-Nitroresorcinol.

Yellow needles from  $CCl_4$ . M.p. 122°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , hot  $C_6H_6$ . Spar. sol.  $CCl_4$ . Prac. insol.  $CS_2$ .  $k = 0.12 \times 10^{-5}$  at 25°. Sublimes. Non-volatile in steam.

*1-Me ether*:  $C_7H_7O_4N$ . MW, 169. Cryst. M.p. 95°. Volatile in steam.

*3-Me ether*: m.p. 144°.

*Di-Me ether*: needles from EtOH. M.p. 76–7° (73°). Insol.  $H_2O$ , ligroin.

*1-Et ether*:  $C_9H_9O_4N$ . MW, 183. Yellow needles. M.p. 79°. Sol. EtOH,  $Et_2O$ , AcOH. Volatile in steam.

*3-Et ether*: needles or plates from EtOH or AcOH. M.p. 131°. Non-volatile in steam.

*Di-Et ether*: m.p. 85°.

*Diacetyl*: plates from EtOH. M.p. 90–1°.

*Dibenzoyl*: m.p. 110°.

Weselsky, Benedikt, *Monatsh.*, 1880, **1**, 892.

Blanksma, *Rec. trav. chim.*, 1902, **21**, 322.

Kauffmann, Kugel, *Ber.*, 1911, **44**, 755.

Holleman, de Mooy, *Rec. trav. chim.*, 1916, **35**, 15.

## 5-Nitroresorcinol.

Cryst. from  $H_2O$ . M.p. 158°.

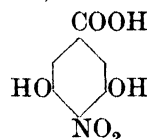
*Me ether*: yellow cryst. M.p. 141–2°.

*Di-Me ether*: yellow needles from AcOEt. M.p. 89°.

*Et ether*: cryst. from  $H_2O$ . M.p. 80°.

Blanksma, *Rec. trav. chim.*, 1908, **27**, 27.

Vermeulen, *Rec. trav. chim.*, 1906, **25**, 26.

4-Nitro- $\alpha$ -resorcylic Acid (4-Nitro-3:5-dihydroxybenzoic acid)

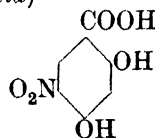
$C_7H_5O_6N$

MW, 199

Dark red needles from  $H_2O$ . Decomp. at 212°. Sol. hot  $H_2O$ , EtOH,  $Et_2O$ .  $FeCl_3 \rightarrow$  reddish-brown col.

*Di-Me ether*: see 4-Nitro-3:5-dimethoxybenzoic Acid.

Meyer, *Monatsh.*, 1887, **8**, 430.

5-Nitro- $\beta$ -resorcylic Acid (5-Nitro-2:4-dihydroxybenzoic acid)

$C_7H_5O_6N$

MW, 199

Yellow leaflets or needles +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p. 215°.

*4-Me ether*:  $C_8H_7O_6N$ . MW, 213. Needles from  $H_2O$ . M.p. about 230° decomp. Spar. sol.  $H_2O$ .  $FeCl_3 \rightarrow$  blood-red col.

*Me ester*:  $C_8H_7O_6N$ . MW, 213. Leaflets from MeOH. M.p. 167°. Sol. hot EtOH. Spar. sol.  $H_2O$ .

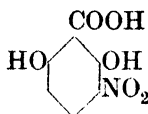
*Nitrile*:  $C_7H_4O_4N_2$ . MW, 180. Yellow needles from AcOH. M.p. 220°. Sol. EtOH, AcOH. Less sol.  $C_6H_6$ . *Diacetyl*: needles from EtOH. M.p. 119°. Sol. AcOH.

*Acetyl*: cryst. M.p. 150°, solidifies and remelts at 175°. Easily decomp.

Lindemann, Könitzer, Romanoff, *Ann.*, 1927, **456**, 292.

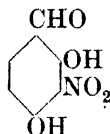
v. Hemmelmayr, *Monatsh.*, 1904, **25**, 25; 1905, **26**, 185.

Gilbody, Perkin, *J. Chem. Soc.*, 1902, **81**, 1056.

**3-Nitro- $\gamma$ -resorcylic Acid** (3-Nitro-2 : 6-dihydroxybenzoic acid) $C_7H_5O_6N$ 

MW, 199

*Nitrile*:  $C_7H_4O_4N_2$ . MW, 180. *6-Me ether*:  $C_8H_6O_4N_2$ . MW, 194. M.p. 163°. *Di-Me ether*: see under 3-Nitro-2 : 6-dimethoxybenzoic Acid. *6-Et ether*:  $C_9H_8O_4N_2$ . MW, 208. M.p. 129°. *6-Me-2-Et ether*:  $C_{10}H_{10}O_4N_2$ . MW, 222. M.p. 60°. *2-Me-6-Et ether*: m.p. 90°. *Di-Et ether*:  $C_{11}H_{12}O_4N_2$ . MW, 236. M.p. 66°.

Blanksma, *Chem. Zentr.*, 1912, II, 339.**3-Nitro- $\beta$ -resorcylic Aldehyde** (3-Nitro-2 : 4-dihydroxybenzaldehyde) $C_7H_5O_5N$ 

MW, 183

*4-Me ether*:  $C_8H_7O_5N$ . MW, 197. Needles from EtOH. M.p. 146-7°. Gives blood-red col. with  $FeCl_3$  in MeOH. *Oxime*: needles. M.p. 191-2°. *Phenylhydrazone*: golden-yellow leaflets. M.p. 180°.

*Di-Me ether*: see 3-Nitro-2 : 4-dimethoxybenzaldehyde.

*4-Me-2-Et ether*:  $C_{10}H_{11}O_5N$ . MW, 225. Cryst. M.p. 57-8°.

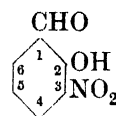
Srikantia, Iyengar, Santanam, *J. Chem. Soc.*, 1932, 526.**5-Nitro- $\beta$ -resorcylic Aldehyde** (5-Nitro-2 : 4-dihydroxybenzaldehyde).

Yellowish-brown prisms from  $C_6H_6$ . M.p. 148-9°.

*4-Me ether*: needles from EtOH or  $CHCl_3$ . M.p. 168-9°. Gives red col. with  $FeCl_3$ . *Oxime*: yellow. M.p. 215-16°. *Phenylhydrazone*: orange. M.p. 197-8°.

*Di-Me ether*: see 5-Nitro-2 : 4-dimethoxybenzaldehyde.

*4-Me-2-Et ether*: cryst. M.p. 138-9°. Mod. sol. AcOH,  $CHCl_3$ . Spar. sol. alcohols. *Oxime*: m.p. 185-6°.

Gattermann, *Ann.*, 1907, 357, 337.Rao, Srikantia, Iyengar, *J. Chem. Soc.*, 1925, 127, 558.**3-Nitrosalicylaldehyde** $C_7H_5O_4N$ 

MW, 167

Needles from AcOH.Aq. M.p. 109-10°.

*Me ether*:  $C_8H_7O_4N$ . MW, 181. Yellow prisms from EtOH.Aq. M.p. 102°.

Miller, *Ber.*, 1887, 20, 1928.**5-Nitrosalicylaldehyde.**

Cryst. from AcOH.Aq. M.p. 126°.

*Me ether*:  $C_8H_7O_4N$ . MW, 181. Needles from  $H_2O$ . M.p. 89-90°. Sol. EtOH,  $Et_2O$ . *Oxime*: m.p. 183°.

*Et ether*:  $C_9H_9O_4N$ . MW, 195. Yellowish needles from EtOH.Aq. M.p. 71-2°. *Semicarbazone*: golden-yellow prisms from EtOH. M.p. 234-5° (223°) decomp. *Azine*: leaflets from AcOH. M.p. 284-5°. *Phenylhydrazone*: red needles from EtOH. M.p. 203-4°.

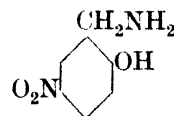
*Oxime*: m.p. 225°.

*Anil*: yellow needles from  $C_6H_6$ . M.p. 133°. Sol.  $CHCl_3$ . Mod. sol.  $C_6H_6$ , AcOH. Spar. sol. MeOH, EtOH,  $Et_2O$ , ligroin.

Miller, *Ber.*, 1887, 20, 1929.Gattermann, *Ann.*, 1912, 393, 225.**6-Nitrosalicylaldehyde.**

Pale yellow prisms from MeOH or pet. ether. M.p. 54-5°.  $FeCl_3 \rightarrow$  reddish-brown col.

*Me ether*: cryst. M.p. 111°. Aq. NaOH added to  $Me_2CO$  sol. gives deep blue ppt.

Ashley, Perkin, Robinson, *J. Chem. Soc.*, 1930, 395.**5-Nitrosalicylamine** (5-Nitro-o-hydroxybenzylamine) $C_7H_8O_3N_2$ 

MW, 168

Yellow leaflets or needles from  $H_2O$ . M.p. 253° decomp.  $HNO_2 \rightarrow$  5-nitrosaligenin.

*B.HCl*: yellowish needles. M.p. 250°.

*N-Di-Et*:  $C_{11}H_{16}O_3N_2$ . MW, 224. Yellow needles from EtOH.Aq. M.p. 68-9°. *B.HCl*: cryst. from EtOH. M.p. 197° decomp.

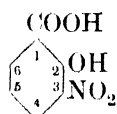
*N-Chloroacetyl*: yellow needles from  $Me_2CO$ . M.p. 185-6°.

*N-Benzoyl*: yellowish needles from  $\text{Me}_2\text{CO}$ . M.p. 217–18°.

Einhorn *et al.*, *Ann.*, 1905, **343**, 243; D.R.P., 156,398, (*Chem. Zentr.*, 1905, I, 55).

Tscherniac, D.R.P., 134,979, (*Chem. Zentr.*, 1902, II, 1084).

### 3-Nitrosalicylic Acid



$\text{C}_7\text{H}_5\text{O}_5\text{N}$

MW, 183

Needles from AcOH. M.p. 148–9° (144°). Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>.  $k = 1.57 \times 10^{-2}$  at 25°.

*Me ether*:  $\text{C}_8\text{H}_7\text{O}_5\text{N}$ . MW, 197. Needles from H<sub>2</sub>O or EtOH. M.p. 194–5° (191–2°).

*Et ether*: 3-nitro-2-ethoxybenzoic acid.  $\text{C}_9\text{H}_9\text{O}_5\text{N}$ . MW, 211. Needles from H<sub>2</sub>O. M.p. 96–7°. *Et ester*:  $\text{C}_{11}\text{H}_{13}\text{O}_5\text{N}$ . MW, 239. Pale yellow oil. B.p. 175°/40 mm. Insol. H<sub>2</sub>O.

*Me ester*:  $\text{C}_8\text{H}_7\text{O}_5\text{N}$ . MW, 197. Needles from MeOH. M.p. 132° (94°).

*Et ester*:  $\text{C}_9\text{H}_9\text{O}_5\text{N}$ . MW, 211. Plates or needles from EtOH. M.p. 48·5° (44°). Spar. sol. H<sub>2</sub>O, EtOH.

*Phenyl ester*: see  $\beta$ -Nitrosalol.

*Chloride*:  $\text{C}_7\text{H}_4\text{O}_4\text{NCl}$ . MW, 201·5. Plates from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 59–61°.

*Amide*:  $\text{C}_7\text{H}_6\text{O}_4\text{N}_2$ . MW, 182. Needles from EtOH, C<sub>6</sub>H<sub>6</sub> or H<sub>2</sub>O. M.p. 155° (145–6°). FeCl<sub>3</sub> → bluish-red col.

*Nitrile*: 6-nitro-2-cyanophenol.  $\text{C}_7\text{H}_4\text{O}_3\text{N}_2$ . MW, 164. Golden-yellow prisms from 90% EtOH. M.p. 132–3°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Cousin, Volmar, *Compt. rend.*, 1914, **159**, 330.

Simonsen, Rau, *J. Chem. Soc.*, 1917, **111**, 224.

Zacharias, *J. prakt. Chem.*, 1891, **43**, 435.

Hübner, *Ann.*, 1879, **195**, 34.

Anschütz, Weber, *Ann.*, 1906, **346**, 338.

Meldrum, Hirve, *J. Indian Chem. Soc.*, 1928, **5**, 95.

Fishman, *J. Am. Chem. Soc.*, 1920, **42**, 2296.

### 4-Nitrosalicylic Acid.

Yellow needles from H<sub>2</sub>O or EtOH.Aq. M.p. 235° (226°). Sol. boiling H<sub>2</sub>O, EtOH, AcOH. Mod. sol. CHCl<sub>3</sub>. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. Bitter taste. FeCl<sub>3</sub> → red col.

*Me ether*:  $\text{C}_8\text{H}_7\text{O}_5\text{N}$ . MW, 197. Needles + H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 147°. *Me ester*:  $\text{C}_9\text{H}_9\text{O}_5\text{N}$ . MW, 211. Needles from MeOH. M.p. 88–9°.

*Phenyl ether*: see 4-Nitro-*o*-phenoxybenzoic Acid.

*Et ester*:  $\text{C}_9\text{H}_9\text{O}_5\text{N}$ . MW, 211. Needles from EtOH. M.p. 87°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. Alc. FeCl<sub>3</sub> → red col. Bitter taste.

*Amide*:  $\text{C}_7\text{H}_6\text{O}_4\text{N}_2$ . MW, 182. Yellowish needles from EtOH-AcOH. M.p. 192–4°.

*Nitrile*: 5-nitro-2-cyanophenol.  $\text{C}_7\text{H}_4\text{O}_3\text{N}_2$ . MW, 164. Yellowish needles + H<sub>2</sub>O from MeOH.Aq. M.p. anhyd. 160–1°. Very sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. hot H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Hygroscopic. *Acetyl*: yellowish needles from MeOH. M.p. 100°. *Benzoyl*: cryst. from EtOH. M.p. 122°.

Borsche, *Ann.*, 1912, **390**, 10.

Simonsen, Rau, *J. Chem. Soc.*, 1917, **111**, 232.

Ullmann, Wagner, *Ann.*, 1907, **355**, 360.

### 5-Nitrosalicylic Acid.

Needles from H<sub>2</sub>O. M.p. 229–30° (227°). Very sol. EtOH. Sol. hot H<sub>2</sub>O.  $k = 8.9 \times 10^{-3}$  at 25°. FeCl<sub>3</sub> → bluish-red col. Heat with lime → *p*-nitrophenol. Zn + AcOH → 5-acetylaminosalicylic acid.

*Me ether*:  $\text{C}_8\text{H}_7\text{O}_5\text{N}$ . MW, 197. Plates or leaflets from H<sub>2</sub>O. M.p. 161°. Sol. EtOH, Me<sub>2</sub>CO, hot H<sub>2</sub>O, AcOEt. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Sublimes. *Me ester*:  $\text{C}_9\text{H}_9\text{O}_5\text{N}$ . MW, 211. Needles from EtOH. M.p. 99–100°. *Nitrile*:  $\text{C}_7\text{H}_4\text{O}_3\text{N}_2$ . MW, 178. Cryst. from H<sub>2</sub>O. M.p. 126°. Sol. hot EtOH. *Benzoyl*: prisms from EtOH. M.p. 117–18°.

*Et ether*: 5-nitro-2-ethoxybenzoic acid.  $\text{C}_9\text{H}_9\text{O}_5\text{N}$ . MW, 211. Plates or prisms from H<sub>2</sub>O. M.p. 163°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. *Et ester*:  $\text{C}_{11}\text{H}_{13}\text{O}_5\text{N}$ . MW, 239. Needles from EtOH. M.p. 98° (68°). *Nitrile*:  $\text{C}_7\text{H}_4\text{O}_3\text{N}_2$ . MW, 192. Cryst. from boiling H<sub>2</sub>O. M.p. 101°.

*Phenyl ether*: see 5-Nitro-*o*-phenoxybenzoic Acid.

*o-Tolyl ether*: 4-nitro-2'-methyldiphenyl ether 2-carboxylic acid. Leaflets. M.p. 187–8°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

*m-Tolyl ether*: 4-nitro-3'-methyldiphenyl ether 2-carboxylic acid. Cryst. M.p. 172°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

*p-Tolyl ether*: 4-nitro-4'-methyldiphenyl ether 2-carboxylic acid. Cryst. M.p. 165°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

*Me ester*:  $C_8H_7O_5N$ . MW, 197. Cryst. from  $Et_2O$ . M.p. 119°.

*Et ester*:  $C_9H_9O_5N$ . MW, 211. Needles from  $EtOH$ . M.p. 102° (96°). Sol.  $EtOH$ ,  $Et_2O$ . Insol.  $H_2O$ .

*Phenyl ester*: see  $\alpha$ -Nitrosalol.

*2-Naphthyl ester*: leaflets from  $EtOH$ . M.p. 201°.

*Amide*:  $C_7H_6O_4N_2$ . MW, 182. Needles from  $EtOH$ . M.p. 225°. Sol. hot  $H_2O$ ,  $EtOH$ .  $FeCl_3 \rightarrow$  bluish-red col.

*Nitrile*: 4-nitro-2-cyanophenol.  $C_7H_4O_3N_2$ . MW, 164. Yellow needles from  $H_2O$ . M.p. 194-6°.

Blanksma, *Chem. Zentr.*, 1908, II, 1827.

Hübner, *Ann.*, 1879, 195, 15.

Thieme, *J. prakt. Chem.*, 1891, 43, 469.

Hirsch, *Ber.*, 1900, 33, 3239.

Simonsen, Rau, *J. Chem. Soc.*, 1917, 111, 223.

Meldrum, Hirve, *J. Indian Chem. Soc.*, 1928, 5, 95.

Chattaway, *J. Chem. Soc.*, 1926, 2725.

Kliegl, Hölle, *Ber.*, 1926, 59, 908.

### 6-Nitrosalicylic Acid.

*Me ether*:  $C_8H_7O_5N$ . MW, 197. Needles from  $H_2O$ . M.p. 179-80°. *Amide*:  $C_8H_8O_4N_2$ . MW, 196. Yellowish needles from  $EtOH$ . M.p. 195°. Mod. sol.  $EtOH$ ,  $Me_2CO$ ,  $CHCl_3$ ,  $AcOEt$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ . *Nitrile*:  $C_8H_6O_3N_2$ . MW, 178. Needles from  $CHCl_3$ ,  $Me_2CO$  or  $AcOEt$ . M.p. 171°.

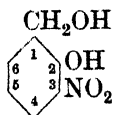
*Et ether*: *amide*.  $C_9H_8O_4N_2$ . MW, 210. M.p. 197°. *Nitrile*:  $C_9H_8O_3N_2$ . MW, 192. Cryst. from  $EtOH$  or  $Me_2CO$ . M.p. 137°. Spar. sol.  $H_2O$ , pet. ether.

*Nitrile*: 3-nitro-2-cyanophenol.  $C_7H_4O_3N_2$ . MW, 164. Yellow leaflets from  $H_2O$ . M.p. 207-8°. Sol.  $EtOH$ ,  $Et_2O$ . Spar. sol.  $H_2O$ .  $C_6H_6$ . Insol. ligroin.

Blanksma, *Chem. Zentr.*, 1908, II, 1826.

De Bruyn, *Rec. trav. chim.*, 1883, 2, 217.

### 3-Nitrosaligenin (3-Nitro-o-hydroxybenzyl alcohol)



$C_7H_7O_4N$  MW, 169

Yellow needles. M.p. 75°. Sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ .

*K salt*: bright red. Spar. sol.  $EtOH$ .

*Me ether*:  $C_8H_9O_4N$ . MW, 183. Needles from  $Et_2O$ -pet. ether. M.p. 42°. Sol.  $Et_2O$ ,

$EtOH$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $AcOH$ . Spar. sol. hot  $H_2O$ .

Fishman, *J. Am. Chem. Soc.*, 1920, 42, 2295.

### 5-Nitrosaligenin (5-Nitro-o-hydroxybenzyl alcohol).

Needles. M.p. 128° (126°).

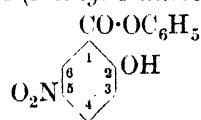
*2-Me ether*: straw coloured needles from  $EtOH$ . M.p. 124-5°. Sol.  $Me_2CO$ . Mod. sol. hot  $H_2O$ ,  $CHCl_3$ . Spar. sol.  $EtOH$ . Conc.  $H_2SO_4 \rightarrow$  orange-brown sol.

*2-Acetyl*: pale brown cryst. from  $C_6H_6$ . M.p. 106.5-108.5° (106-7°). Sol.  $EtOH$ ,  $AcOH$ . Mod. sol.  $C_6H_6$ . Spar. sol. ligroin.

Jacobs, Heidelberg, *J. Biol. Chem.*, 1915, 20, 675.

Bayer, D.R.P., 136,680, (*Chem. Zentr.*, 1902, II, 1439); D.R.P., 148,977, (*Chem. Zentr.*, 1904, I, 699).

### $\alpha$ -Nitrosalol (Phenyl 5-nitrosalicylate)



$C_{13}H_9O_5N$  MW, 259

Needles from  $EtOH$  or  $AcOH$ . M.p. 152°.

*Acetyl*: needles from  $EtOH$ . M.p. 118°.

Knebel, *J. prakt. Chem.*, 1891, 43, 381.

### $\beta$ -Nitrosalol (Phenyl 3-nitrosalicylate).

Prisms from  $EtOH$ . M.p. 102°.

*Acetyl*: m.p. 95°.

See previous reference.

### p-Nitrosoacetanilide.

See under p-Nitrosoaniline.

### N-Nitrosoacetanilide



$C_8H_8O_2N_2$  MW, 164

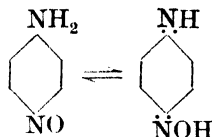
Pale yellow needles from pet. ether. M.p. 50.5-51° decomp. Sol.  $EtOH$ ,  $Et_2O$ ,  $AcOH$ . Sol. in  $Et_2O$  pptes benzene diazonium nitrate. Sol. in ligroin explodes on warming.  $H_2O_2$  in  $Et_2O \rightarrow$  nitrosophenylhydroxylamine.  $Zn + EtOH$  (or  $AcOH$ )  $\rightarrow$  acetanilide.  $NaOH \rightarrow$  sodium benzene diazoate.  $C_6H_6 \rightarrow$  diphenyl + N +  $AcOH$ . Toluene  $\rightarrow$  2- and 4-methyl-diphenyl.  $C_6H_5OH \rightarrow$  2-hydroxyazobenzene. Dry  $HCl \rightarrow$  acetanilide +  $NOCl$ .

Fischer, *Ber.*, 1876, 9, 463.

v. Pechmann, Frobenius, *Ber.*, 1894, 27, 651.

Bamberger, *Ber.*, 1894, 27, 925 (Footnote).

Grieve, Hey, *J. Chem. Soc.*, 1934, 1803.

**p-Nitrosoaniline** (*p*-Benzoquinoneimine oxime)C<sub>6</sub>H<sub>6</sub>ON<sub>2</sub> MW, 122

Steel-blue needles from C<sub>6</sub>H<sub>6</sub>. M.p. 173-4°. Sol. H<sub>2</sub>O with green col. KMnO<sub>4</sub> → *p*-nitroaniline. Sn + HCl → *p*-phenylenediamine. NaOH → NH<sub>3</sub> + *p*-nitrosophenol.

N-Me: see *p*-Nitroso-*N*-methylaniline.N-Di-Me: see *p*-Nitroso-*N*-dimethylaniline.N-Et: see *p*-Nitroso-*N*-ethylaniline.N-Di-Et: see *p*-Nitroso-*N*-diethylaniline.

N-Propyl: C<sub>9</sub>H<sub>12</sub>ON<sub>2</sub>. MW, 164. Steel-blue needles from EtOH.Aq. M.p. 59°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub> with green col. Insol. pet. ether.

N-Dipropyl: C<sub>12</sub>H<sub>18</sub>ON<sub>2</sub>. MW, 206. Green cryst. from ligroin. M.p. 42°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Hydrochloride: decomp. at 160-5°.

N-Butyl: C<sub>10</sub>H<sub>14</sub>ON<sub>2</sub>. MW, 178. Blue needles from EtOH.Aq. M.p. 58-9°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, pet. ether.

N-Dibutyl: C<sub>14</sub>H<sub>22</sub>ON<sub>2</sub>. MW, 234. Green oil. B, HCl: greenish-yellow cryst. from EtOH-Et<sub>2</sub>O. FeCl<sub>3</sub> → red col. B<sub>2</sub>CuCl<sub>2</sub>: green needles from EtOH. M.p. 123-5°. B, ZnCl<sub>2</sub>: m.p. 153° decomp.

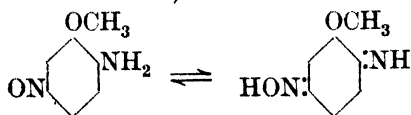
N-Isobutyl: blue cryst. from EtOH. M.p. 93-4°.

N-Acetyl: *p*-nitrosoacetanilide. C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>. MW, 164. Green plates or prisms from EtOH. M.p. 174-5° decomp.

Wacher, *Ann.*, 1888, 243, 291.Fischer, Schäffer, *Ann.*, 1895, 286, 151.Fischer, Hepp, *Ber.*, 1887, 20, 2475.

Reilly, Hickinbottom, *J. Chem. Soc.*, 1917, 111, 1030; 1918, 113, 103.

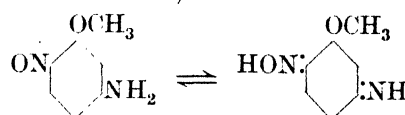
Jacobs, Heidelberg, *J. Biol. Chem.*, 1915, 21, 115.

**5-Nitroso-o-anisidine** (2-Methoxy-*p*-benzoquinoneimine 4-oxime)C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub> MW, 152

Green cryst. from C<sub>6</sub>H<sub>6</sub> or Et<sub>2</sub>O. M.p. 107°. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Sol. acids.

N-Me: C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 166. Green

plates from Et<sub>2</sub>O, blue plates from C<sub>6</sub>H<sub>6</sub>. M.p. 110°. Sol. MeOH, EtOH, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin.

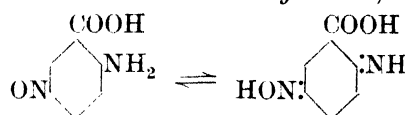
Best, *Ann.*, 1889, 255, 178, 186.**6-Nitroso-m-anisidine** (3-Methoxy-*p*-benzoquinoneimine 4-oxime)C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub> MW, 152

Prisms from EtOH. M.p. 209° decomp.

I.G., D.R.P., 561,424, (*Chem. Zentr.*, 1933, II, 444).

**Nitrosoanisol.**

See under Nitrosophenol.

**5-Nitrosoanthranilic Acid** (*p*-Benzoquinoneimine 4-oxime 2-carboxylic acid)C<sub>7</sub>H<sub>6</sub>O<sub>3</sub>N<sub>2</sub> MW, 166

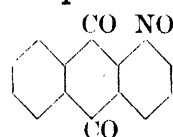
Green cryst. from hot H<sub>2</sub>O. Mod. sol. H<sub>2</sub>O. Insol. Et<sub>2</sub>O.

N-Me: see 5-Nitroso-*N*-methylantranilic Acid.

N-Et: see 5-Nitroso-*N*-ethylantranilic Acid.

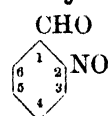
Me ester: C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>. MW, 180. Green needles from boiling H<sub>2</sub>O. M.p. 167-8°. Dichroic. Sublimes in pale green needles.

Et ester: C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 194. Needles from hot H<sub>2</sub>O. M.p. 139°. Spar. sol. EtOH. Dichroic.

Houben, Schreiber, *Ber.*, 1920, 53, 2358.**1-Nitrosoanthraquinone**C<sub>14</sub>H<sub>7</sub>O<sub>3</sub>N MW, 237

Rose coloured needles from EtOH. M.p. 223-4°.

Beisler, Jones, *J. Am. Chem. Soc.*, 1922, 44, 2305.

**o-Nitrosobenzaldehyde**C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>N

MW, 135



Needles from  $\text{CCl}_4$ . M.p. 113–113.5°. Sol. cold  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{AcOH}$ , warm  $\text{EtOH}$ ,  $\text{CCl}_4$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ . Very spar. sol. pet. ether. Dichroic. Volatile in steam with slight decomp.

Bamberger, *Ber.*, 1918, **51**, 624.

### m-Nitrosobenzaldehyde.

Needles. M.p. 106.5–107°. Gives green sols. in usual solvents. Volatile in steam.

Bamberger, *Ber.*, 1895, **28**, 250.

Alway, *Ber.*, 1903, **36**, 2310.

### p-Nitrosobenzaldehyde.

Yellow needles from  $\text{AcOH}$ . M.p. 137–8°. Volatile in steam. Readily polymerised. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  deep violet sol.

Kirpal, *Ber.*, 1897, **30**, 1599.

Alway, *Ber.*, 1903, **36**, 2308.

### Nitrosobenzene

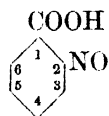
$\text{C}_6\text{H}_5\text{ON}$   $\text{C}_6\text{H}_5\text{NO}$  MW, 107

Cryst. from  $\text{EtOH-Et}_2\text{O}$ . M.p. 67.5–68°. B.p. 57–9°/18 mm. Mod. sol. usual solvents. Very volatile. Alk.  $\text{H}_2\text{O}_2 \rightarrow$  nitrobenzene.  $\text{Sn} + \text{HCl} \rightarrow$  aniline + chloroaniline.  $\text{NaOH.Aq.} \rightarrow$  azoxybenzene.

$\text{B}_5\text{CdI}_2$ : cryst. from  $\text{EtOH}$ . M.p. 114°. Decomp. by  $\text{H}_2\text{O}$ .

Vanino, *Präparative Chemie*, Vol. II, 626.

### o-Nitrosobenzoic Acid



$\text{C}_7\text{H}_5\text{O}_3\text{N}$  MW, 151

Cryst. from  $\text{EtOH}$  or hot  $\text{AcOH}$ . Darkens at 180°, m.p. 210° decomp. (214°). Sol. hot  $\text{EtOH}$ ,  $\text{AcOH}$ . Less sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Sols. are green.

*Me ester*:  $\text{C}_8\text{H}_7\text{O}_3\text{N}$ . MW, 165. Needles. M.p. 156.5–157.5° (152–3°). Sol. conc.  $\text{H}_2\text{SO}_4$  with orange-red col.

*Et ester*:  $\text{C}_9\text{H}_9\text{O}_3\text{N}$ . MW, 179. Cryst. M.p. 120–1°.

*Propyl ester*:  $\text{C}_{10}\text{H}_{11}\text{O}_3\text{N}$ . MW, 193. Prisms from  $\text{C}_6\text{H}_6$  or  $\text{MeOH}$ . M.p. 95°. Sol. boiling  $\text{C}_6\text{H}_6$ . Mod. sol. hot  $\text{MeOH}$ .

*Isopropyl ester*: prisms from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 117–18°. Sol. boiling  $\text{MeOH}$ , hot  $\text{C}_6\text{H}_6$ .

*Isobutyl ester*:  $\text{C}_{11}\text{H}_{13}\text{O}_3\text{N}$ . MW, 207. Needles. M.p. 99–99.5°.

Bamberger, Elger, *Ann.*, 1909, **371**, 339; *Ber.*, 1903, **36**, 3651.

Bamberger, Pyman, *Ber.*, 1909, **42**, 2309, 2326.

Ciamician, Silber, *Ber.*, 1901, **34**, 2044.

Alway, Walker, *Ber.*, 1903, **36**, 2312.

### m-Nitrosobenzoic Acid.

Cryst. Decomp. at 230°. Gives green sols.

*Me ester*: cryst. from  $\text{AcOH}$ . M.p. 93°. Gives bluish-green sols.

*Et ester*: cryst. from  $\text{EtOH}$ . M.p. 52–3°. Gives green sols.

Alway, *Ber.*, 1904, **37**, 334.

Alway, Walker, *Ber.*, 1903, **36**, 2313.

Alway, Gortner, *Am. Chem. J.*, 1904, **32**, 401.

### p-Nitrosobenzoic Acid.

Yellow powder. Decomp. at 250°. Mod. sol. hot  $\text{EtOH}$ . Spar. sol.  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ . Non-volatile in steam.

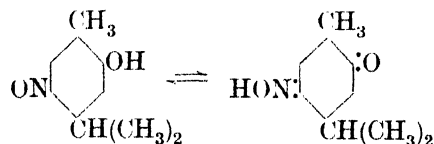
*Me ester*: yellow needles from  $\text{EtOH}$ . M.p. 128.5–129.5°. Sol. hot  $\text{EtOH}$ ,  $\text{AcOH}$ . Gives green sols.

*Et ester*: yellow needles from  $\text{EtOH}$ . M.p. 81°. Sols. are green.

See first two references above and also

Alway, Pinckney, *Am. Chem. J.*, 1904, **32**, 399.

### 5-Nitrosocarvacrol (Thymoquinone 4-oxime)



$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$  MW, 179

Yellowish prisms from  $\text{C}_6\text{H}_6$ , needles from  $\text{EtOH.Aq.}$  M.p. 153°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . Alk.  $\text{K}_3\text{Fe(CN)}_6 \rightarrow$  5-nitrosocarvacrol.  $\text{Sn} + \text{HCl} \rightarrow$  5-amino-carvacrol.

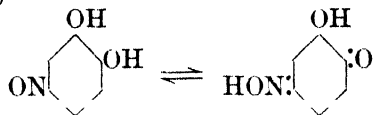
*Phenylsemicarbazone*: yellow needles from  $\text{AcOH}$ . M.p. 204–5°.

Paternò, Canzoneri, *Gazz. chim. ital.*, 1878, **8**, 501.

Mazzara, Plancher, *Gazz. chim. ital.*, 1891, **21**, ii, 155.

Klages, *Ber.*, 1899, **32**, 1518.

**4-Nitrosocatechol** (*Hydroxy-p-benzoquinone 4-oxime*)

C<sub>6</sub>H<sub>5</sub>O<sub>3</sub>N

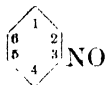
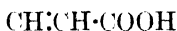
MW, 139

1-*Me ether*: see 5-Nitrosoguaiacol.

1-*Et ether*: C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 167. Needles. Decomp. on heating. Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Sol. alkalis with red col.

Pfob, *Monatsh.*, 1897, **18**, 479.

***m*-Nitrosocinnamic Acid**

C<sub>9</sub>H<sub>7</sub>O<sub>3</sub>N

MW, 177

Needles from EtOH. Decomp. at 230°. Sols. are green.

*Et ester*: C<sub>11</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 205. Green plates from EtOH. M.p. 65–6°. Sol. EtOH.

Alway, Bonner, *Am. Chem. J.*, 1904, **32**, 396.

***p*-Nitrosocinnamic Acid.**

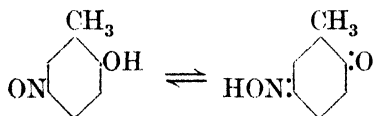
Yellow cryst. powder. Decomp. at 220°. Sol. EtOH, AcOH.

*Me ester*: C<sub>10</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 191. Yellow needles. M.p. 111–12°.

*Et ester*: yellow needles from EtOH. M.p. 72–3°. Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub> with greenish-yellow col.

See previous reference.

**5-Nitroso-*o*-cresol** (*Toluquinone 4-oxime*)

C<sub>7</sub>H<sub>7</sub>O<sub>2</sub>N

MW, 137

Needles from H<sub>2</sub>O. M.p. 134–5° decomp. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Less sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Alk. K<sub>3</sub>Fe(CN)<sub>6</sub> → 5-nitro-*o*-cresol.

*Me ether*: C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 151. Pale yellow prisms from ligroin. M.p. 73–4°. Sol. most solvents.

2:4-Dinitrophenyl ether: m.p. 154°.

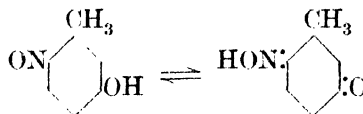
*Acetyl*: exists in two stereoisomeric forms. (i) Thick prisms. M.p. 112–13°. (ii) Cryst.

M.p. 85–7°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, ligroin.

Bridge, Morgan, *Am. Chem. J.*, 1898, **20**, 766.

Nölting, Kohn, *Ber.*, 1884, **17**, 370.

**6-Nitroso-*m*-cresol** (*Toluquinone 1-oxime*)

C<sub>7</sub>H<sub>7</sub>O<sub>2</sub>N

MW, 137

Needles from H<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>, prisms from AcOH. M.p. 156° (165°). Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Less sol. Et<sub>2</sub>O. Mod. sol. hot H<sub>2</sub>O. Aq. sol. reacts acid.  $k = 3.5 \times 10^{-7}$  at 25°. Gives Liebermann nitroso reaction.

*Me ether*: yellow needles from ligroin. M.p. 69°.

*Acetyl*: prisms from EtOH or ligroin. M.p. 92°.

Bridge, Morgan, *Am. Chem. J.*, 1898, **20**, 774.

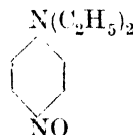
Wurster, Riedel, *Ber.*, 1879, **12**, 1799.

v. Erp, *Rec. trav. chim.*, 1911, **30**, 276.

***N*-Nitrosodiethylamine.**

See Diethylnitrosamine.

***p*-Nitroso-*N*-diethylaniline**

C<sub>10</sub>H<sub>14</sub>ON<sub>2</sub>

MW, 178

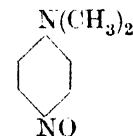
Green prisms from Et<sub>2</sub>O, green leaflets from Me<sub>2</sub>CO. M.p. 87–8° (84°). D<sub>4</sub><sup>25</sup> 1.24. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

Kopp, *Ber.*, 1875, **8**, 621.

***N*-Nitrosodimethylamine.**

See Dimethylnitrosamine.

***p*-Nitroso-*N*-dimethylaniline**

C<sub>8</sub>H<sub>10</sub>ON<sub>2</sub>

MW, 150

Green plates from Et<sub>2</sub>O. M.p. 92.5–93.5° (87.8°).  $k = 1.95 \times 10^{-10}$  at 25°. Heat of comb. C<sub>p</sub> 1123 Cal. Volatile in steam. Readily reduced to *N*-dimethyl-*p*-phenylenediamine. Used as rubber vulcanising accelerator (Accelerene).

*B, HCl*: yellow needles. M.p. 177° decomp.  
Heat of comb.  $C_p$  1120.8 Cal.

*B, HBr*: yellow cryst. Decomp. at 207°.

*B, HNO<sub>3</sub>*: m.p. 157–8° decomp.

*Trichloroacetate*: yellow cryst. M.p. 103°.

*Picrate*: yellow cryst. Decomp. at 140°.  
Sol. EtOH. Spar. sol. CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>.  
Insol. Et<sub>2</sub>O.

Baeyer, Caro, *Ber.*, 1874, 7, 810, 963.

Vanino, *Präparative Chemie*, Vol. II, 629.

### Nitroso-*N*-dimethyltoluidine.

See under Nitrosotoluidine.

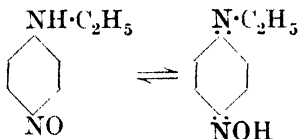
### *N*-Nitrosodiphenylamine.

See under Diphenylamine.

### *p*-Nitroso-*N*-dipropylaniline.

See under *p*-Nitrosoaniline.

***p*-Nitroso-*N*-ethylaniline** (*p*-Benzoquinone-ethylimine oxime)



$C_8H_{10}ON_2$

MW, 150

Green plates from C<sub>6</sub>H<sub>6</sub>. M.p. 78°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. Warm NaOH → *p*-nitrosophenol + ethylamine.

*Oxalate*: m.p. 124°.

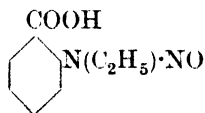
*B<sub>3</sub>, AgNO<sub>3</sub>*: dark green cryst. from EtOH. Aq. M.p. 121° decomp.

*Picrate*: yellow needles. M.p. 131°.

Fischer, Hepp, *Ber.*, 1886, 19, 2993.

Fischer, *Ann.*, 1895, 286, 156.

### *N*-Nitroso-*N*-ethylanthrnic Acid



$C_9H_{10}O_3N_2$

MW, 194

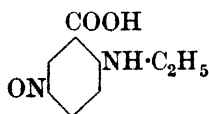
Needles. M.p. 90–1°.

*Amide*:  $C_9H_{11}O_2N_3$ . MW, 193. Needles from H<sub>2</sub>O. M.p. 110°.

Vorländer, v. Schilling, Schrödter, *Ber.*, 1901, 34, 1645.

Finger, *J. prakt. Chem.*, 1888, 37, 442.

### 5-Nitroso-*N*-ethylanthrnic Acid



$C_9H_{10}O_3N_2$

MW, 194

Green needles from H<sub>2</sub>O, pale green prisms from C<sub>6</sub>H<sub>6</sub>. Decomp. at 142–52°. Sol. EtOH, Et<sub>2</sub>O.

*Me ester*:  $C_{10}H_{12}O_3N_2$ . MW, 208. Green needles from ligroin. M.p. 91°.

*Et ester*:  $C_{11}H_{14}O_3N_2$ . MW, 222. Green cryst. from ligroin. M.p. 87–8°.

Houben, Brassert, Ettinger, *Ber.*, 1909, 42, 2752.

Houben, *ibid.*, 3195.

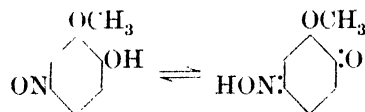
### Nitroso-*N*-ethylnaphthylamine.

See under Nitrosomethylaniline.

### Nitroso-*N*-ethyltoluidine.

See under Nitrosotoluidine.

**5-Nitrosoguaiacol** (2-Methoxy-*p*-benzoquinone 4-oxime)



$C_7H_7O_3N$

MW, 153

Yellow leaflets from CHCl<sub>3</sub>. Decomp. at 165°. Sol. EtOH, CHCl<sub>3</sub>. Less sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

*Me ether*: 4-nitrosoveratrol.  $C_8H_9O_3N$ . MW, 167. Yellowish-white cryst. M.p. 105–6°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

*Acetyl*: cryst. M.p. 156–8° decomp.

*Benzoyl*: yellow cryst. from EtOH. M.p. 188°. Sol. CHCl<sub>3</sub>, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin. Insol. Et<sub>2</sub>O, CS<sub>2</sub>.

Pfob, *Monatsh.*, 1897, 18, 472.

Best, *Ann.*, 1889, 255, 184.

Bridge, Morgan, *Am. Chem. J.*, 1899, 22, 486.

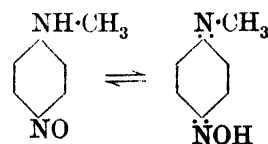
### Nitrosohydroxyquinoline.

See under Quinolinequinone.

### *N*-Nitrosomethylaniline.

See under Methylaniline.

***p*-Nitroso-*N*-methylaniline** (*p*-Benzoquinone-methylimine oxime)



$C_7H_8ON_2$

MW, 136

Bluish plates from C<sub>6</sub>H<sub>6</sub>. M.p. 118°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Less sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, ligroin. Possesses acid and basic properties.  $k$  (acid) =  $1.12 \times 10^{-13}$  at 6°;  $k$  (base) =  $1.63 \times 10^{-10}$  at 25°.

**N-Et**:  $C_9H_{12}ON_2$ . MW, 164. Green plates from EtOH.Aq. M.p. 66–7°.

**N-β-Chloroethyl**:  $C_9H_{11}ON_2Cl$ . MW, 198.5. Green leaflets from EtOH–pet. ether. M.p. 69°.

**N-β-Bromoethyl**:  $C_9H_{11}ON_2Br$ . MW, 243. Green cryst. from EtOH–pet. ether. M.p. 70°.

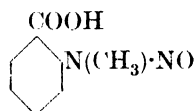
**N-Propyl**:  $C_{10}H_{14}ON_2$ . MW, 178. *B.HCl*: brownish-green leaflets from Et<sub>2</sub>O. M.p. 105°. Sol. H<sub>2</sub>O, EtOH. Very hygroscopic.

Fischer, Hepp, *Ber.*, 1886, **19**, 2991.

Cain, *Chem. Zentr.*, 1911, **1**, 1742.

Stoermer, v. Lepel, *Ber.*, 1896, **29**, 2112.

**N-Nitroso-N-methylantranilic Acid**



$C_8H_8O_3N_2$  MW, 180

Prisms from  $C_6H_6$ . M.p. 128°. Very sol. EtOH, Et<sub>2</sub>O. Mod. sol.  $CHCl_3$ , AcOH. Spar. sol. ligroin.

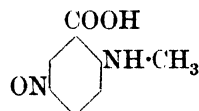
**Me ester**:  $C_9H_{10}O_3N_2$ . MW, 194. Pale yellow oil. B.p. 176–7°/12 mm.  $D_4^{18.8}$  1.2107.  $n_D^{18.8}$  1.55219.

**Amide**:  $C_8H_9O_2N_3$ . MW, 179. Needles from EtOH. M.p. 149°.

Fortmann, *J. prakt. Chem.*, 1893, **47**, 400; 1897, **55**, 126.

Zacharias, *J. prakt. Chem.*, 1891, **43**, 449.

**5-Nitroso-N-methylantranilic Acid**



$C_8H_8O_3N_2$  MW, 180

Olive-green cryst. from EtOH or AcOH. Insol. Et<sub>2</sub>O, pet. ether. Dil. NaOH on standing → 5-nitrososalicylic acid.

**Me ester**:  $C_9H_{10}O_3N_2$ . MW, 194. Light green needles from pet. ether–ligroin. M.p. 119°. **N-Carbomethoxyl**: green needles from MeOH. M.p. 164–5°. Sol. warm  $C_6H_6$ . Insol. pet. ether, ligroin. **N-Carboethoxyl**: green needles from EtOH. M.p. 125° decomp.

**Et ester**:  $C_{10}H_{12}O_3N_2$ . MW, 208. Green needles from EtOH. M.p. 89°. Sol. most solvents. Mod. sol. hot H<sub>2</sub>O. **N-Carboethoxyl**: needles from EtOH. M.p. 131° decomp.

**Phenyl ester**:  $C_{14}H_{12}O_3N_2$ . MW, 256. Green needles from ligroin. M.p. 135–6°.

**N-Carbomethoxyl**: light green. Decomp. at 115–16°.

**N-Carboethoxyl**: green needles. M.p. 115–16° decomp.

Houben, Schreiber, *Ber.*, 1920, **53**, 2351.

Houben, Brassert, *Ber.*, 1907, **40**, 4740.

Houben, Brassert, Ettinger, *Ber.*, 1909, **42**, 2751.

Riedel, D.R.P., 256,461, (*Chem. Zentr.*, 1913, **I**, 866).

**Nitroso-N-methylnaphthylamine.**

See under *N-Methyl-2-naphthylamine and Nitrosomethylnaphthylamine.*

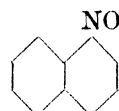
**Nitrosomethyltoluidine.**

See under *N-Methyltoluidine and Nitrosomethyltoluidine.*

**Nitrosomethylurea.**

See under *Methylurea.*

**1-Nitrosomethylurea**

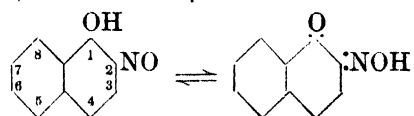


$C_{10}H_7ON$  MW, 157

Pale yellow cryst. from Me<sub>2</sub>CO. M.p. 98°. Decomp. about 134°. Sol. AcOEt,  $CHCl_3$ . Mod. sol. EtOH, Et<sub>2</sub>O,  $C_6H_6$ . Spar. sol. pet. ether. Decomp. by warm alkalis and acids. Phenol + conc. H<sub>2</sub>SO<sub>4</sub> → blue col.

Willstätter, Kubli, *Ber.*, 1908, **41**, 1938.

**2-Nitroso-1-naphthol (1 : 2-Naphthoquinone 2-oxime)**



$C_{10}H_7O_2N$  MW, 173

Yellow needles from H<sub>2</sub>O or  $C_6H_6$ . M.p. 162–4°. Sol. MeOH, EtOH, AcOH, Me<sub>2</sub>CO. Less sol. Et<sub>2</sub>O,  $C_6H_6$ ,  $CHCl_3$ , CS<sub>2</sub>, pet. ether. Insol. cold H<sub>2</sub>O. Volatile in steam. Conc. H<sub>2</sub>SO<sub>4</sub> → intense red sol.

**Me ether**:  $O:C_{10}H_6:N\cdot OCH_3$ . MW, 187. Yellowish-green needles from EtOH.Aq. M.p. 95°. Sol. EtOH. Conc. H<sub>2</sub>SO<sub>4</sub> → intense red sol.

**Et ether**:  $O:C_{10}H_6:N\cdot OC_2H_5$ . MW, 201. Greenish-yellow needles. M.p. 101°. Sol. EtOH.

**Benzoyl**:  $O:C_{10}H_6:N\cdot O\cdot CO\cdot C_6H_5$ . Yellow needles from Me<sub>2</sub>CO– $CHCl_3$  or AcOEt. M.p. 189–90° (162°). Sol.  $CHCl_3$ , AcOH, AcOEt.

Less sol.  $C_6H_6$ ,  $CS_2$ . Spar. sol. EtOH,  $H_2O$ ,  $Et_2O$ , ligroin.

Fuchs, *Ber.*, 1875, **8**, 626.

Reverdin, de la Harpe, *Ber.*, 1893, **26**, 1280.

Meisenheimer, Witte, *Ber.*, 1903, **36**, 4169.

**4-Nitroso-1-naphthol** (1 : 4-Naphthoquinone-oxime).

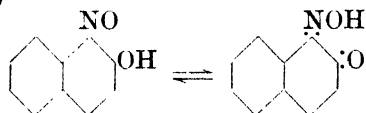
Pale yellow needles from  $C_6H_6$ . M.p.  $198^\circ$  ( $193^\circ$ ). Sol. EtOH, MeOH,  $Me_2CO$ ,  $Et_2O$ . Spar. sol.  $C_6H_6$ ,  $CHCl_3$ ,  $CS_2$ . Sol. alkalis.

*Me ether*:  $O:C_{10}H_6:N\cdot OCH_3$ . MW, 187. Pale yellow needles from EtOH,  $Et_2O$  or MeOH. M.p.  $85^\circ$  ( $80-2^\circ$ ). Sol. most solvents. Conc.  $H_2SO_4 \rightarrow$  yellow sol.

Meyer, Lenhardt, *Ann.*, 1913, **398**, 79.

Meisenheimer, *Ann.*, 1907, **355**, 305.

**1-Nitroso-2-naphthol** (1 : 2-Naphthoquinone 1-oxime)



$C_{10}H_7O_2N$  MW, 173

Orange prisms or plates from EtOH or  $C_6H_6$ . M.p.  $109.5^\circ$ . Very sol.  $C_6H_6$ ,  $CS_2$ , AcOH. Spar. sol.  $H_2O$ , pet. ether. Sol. 42 parts EtOH at  $13^\circ$ . Used as reagent for cobalt.

*Me ether*:  $O:C_{10}H_6:N\cdot OCH_3$ . MW, 187. Yellow prisms from ligroin. M.p.  $75^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Mod. sol. hot  $H_2O$ . Spar. sol. ligroin. Conc.  $H_2SO_4 \rightarrow$  deep red sol.

*Et ether*:  $O:C_{10}H_6:N\cdot OC_2H_5$ . MW, 201. Needles from EtOH.Aq. or ligroin. M.p.  $50-60^\circ$ . Spar. volatile in steam.

*Benzyl ether*:  $O:C_{10}H_6:N\cdot O\cdot CH_2\cdot C_6H_5$ . Pale yellow cryst. from EtOH-pet. ether. M.p.  $101^\circ$  ( $98^\circ$ ).

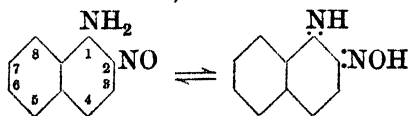
*Benzoyl*:  $O:C_{10}H_6:N\cdot O\cdot CO\cdot C_6H_5$ . Yellow cryst. from EtOH. M.p.  $114^\circ$ . Sol. EtOH, AcOH. Spar. sol.  $H_2O$ , ligroin.

*Benzenesulphonyl*: yellow prisms from  $Me_2CO$ . M.p.  $124-5^\circ$  decomp. Above m.p.  $\rightarrow$  isomer, m.p.  $141^\circ$ .

Lagodzinski, Hardine, *Ber.*, 1894, **27**, 3076.

Marvel, Porter, *Organic Syntheses*, Collective Vol. I, 403.

**2-Nitroso-1-naphthylamine** (1 : 2-Naphthoquinoneimine 2-oxime)



$C_{10}H_8ON_2$  MW, 172

Green prisms from  $C_6H_6$ . Sol. EtOH,  $C_6H_6$ . Less sol.  $Et_2O$ ,  $CHCl_3$ . Spar. sol. hot  $H_2O$ , ligroin. Alk.  $K_3Fe(CN)_6 \rightarrow \alpha : \beta$ -naphthafurazan.

*N-Et*:  $C_{12}H_{12}ON_2$ . MW, 200. Green leaflets +  $H_2O$  from EtOH.Aq. M.p.  $95^\circ$ .

Harden, *Ann.*, 1889, **255**, 162.

**4-Nitroso-1-naphthylamine** (1 : 4-Naphthoquinoneimine 4-oxime).

*N-Me*:  $C_{11}H_{10}ON_2$ . MW, 186. Golden cryst. from  $C_6H_6$ . M.p.  $157^\circ$  decomp. Mod. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*N-Et*:  $C_{12}H_{12}ON_2$ . MW, 200. Cryst. from  $C_6H_6$ . M.p.  $133^\circ$  decomp. Sol. EtOH,  $C_6H_6$ ,  $CHCl_3$ . Insol. ligroin. *Picrate*: green plates. M.p.  $174^\circ$  decomp.

*N-Di-Et*:  $C_{14}H_{16}ON_2$ . MW, 228. Golden-red cryst. from EtOH. M.p.  $165^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  dark blue sol.

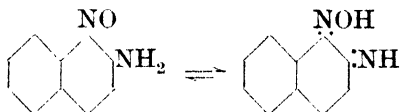
*N-Phenyl*:  $C_{16}H_{12}ON_2$ . MW, 248. Yellowish-brown leaflets or plates from MeOH.Aq. M.p.  $150^\circ$ .

Fischer, Apitsch, *Ann.*, 1895, **286**, 160.

Kock, *Ann.*, 1888, **243**, 310.

I.G., F.P., 701,915, (*Chem. Abstracts*, 1931, **25**, 4135).

**1-Nitroso-2-naphthylamine** (1 : 2-Naphthoquinoneimine 1-oxime)



$C_{10}H_8ON_2$  MW, 172

Green needles from EtOH.Aq. M.p.  $150-2^\circ$ . Sol. hot  $H_2O$ , org. solvents. Mild ox. agents  $\rightarrow \alpha : \beta$ -naphthafurazan.

*N-Me*:  $C_{11}H_{10}ON_2$ . MW, 186. Dark green prisms or leaflets from MeOH. M.p.  $148-9^\circ$  decomp. *B,HCl*: yellow needles from EtOH- $Et_2O$ . *Picrate*: yellow needles from EtOH.Aq. Mod. sol. EtOH,  $C_6H_6$ . Spar. sol.  $H_2O$ . *Acetyl*: yellow prisms from  $C_6H_6$ . M.p.  $140-1^\circ$  decomp.

*N-Et*:  $C_{12}H_{12}ON_2$ . MW, 200. Green prisms or plates from MeOH or  $C_6H_6$ -ligroin. M.p.  $120-1^\circ$ . *N-Acetyl*: yellow leaflets from EtOH.Aq. Decomp. at  $116-18^\circ$ .

*N-Propyl*:  $C_{13}H_{14}ON_2$ . MW, 214. Dark green prisms from EtOH. M.p.  $115^\circ$  decomp. Sol.  $C_6H_6$ . Mod. sol. EtOH. Spar. sol.  $H_2O$ . *B,HCl*: yellow needles from dil. HCl. Decomp. at  $280^\circ$ . *Picrate*: yellow needles from EtOH. Decomp. at  $235^\circ$ . *Acetyl*: yellow leaflets from EtOH.Aq. M.p.  $114^\circ$ .

*N-Butyl*:  $C_{14}H_{16}ON_2$ . MW, 228. Dark green needles from MeOH, prisms from EtOH. M.p. 98–9°. *Picrate*: yellow needles. Decomp. at 244°. *Acetyl*: leaflets from Et<sub>2</sub>O. Turns yellow in air.

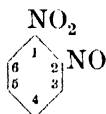
*N-Isoamyl*:  $C_{15}H_{18}ON_2$ . MW, 242. Deep green leaflets or needles from MeOH. M.p. 82°.

*N-Acetyl*: yellow cryst. powder from pet. ether. Decomp. about 136°.

Ijinski, *Ber.*, 1884, **17**, 391.

Fischer, Dietrich, Weiss, *J. prakt. Chem.*, 1920, **100**, 167.

### o-Nitrosnitrobenzene



$C_6H_4O_3N_2$

MW, 152

Yellowish cryst. from AcOEt or Me<sub>2</sub>CO. M.p. 126–126.5° after turning green at 120°. Sol. hot CHCl<sub>3</sub>, hot Me<sub>2</sub>CO, hot C<sub>6</sub>H<sub>6</sub>. Mod. sol. hot EtOH, hot ligroin. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O, pet. ether. Sols. are deep green. Alc. sol. with alkali → bluish-violet col. Volatile in steam. HNO<sub>3</sub> (D 1.26) → o-dinitrobenzene.

Bamberger, Hübner, *Ber.*, 1903, **36**, 3803.

Meisenheimer, Patzig, *Ber.*, 1906, **39**, 2530.

### m-Nitrosnitrobenzene.

Needles. M.p. 90–1° → a green liq. Sol. CHCl<sub>3</sub>, Me<sub>2</sub>CO, AcOH, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Spar. sol. Et<sub>2</sub>O. Prac. insol. pet. ether. Volatile in steam.

Bamberger, Hübner, *Ber.*, 1903, **36**, 3806.

Alway, Gortner, *Ber.*, 1905, **38**, 1900.

Brand, *ibid.*, 4011.

### p-Nitrosnitrobenzene.

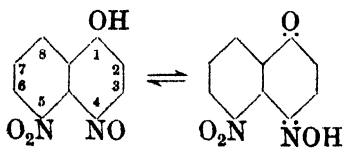
Pale yellow needles from EtOH. M.p. 118.5–119° → a green liq. Sol. with green col. in CHCl<sub>3</sub>, AcOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Spar. sol. Et<sub>2</sub>O, ligroin. Prac. insol. H<sub>2</sub>O. Volatile in steam. HNO<sub>3</sub> (D 1.26) → p-dinitrobenzene. Alc. sol. with alkali → red col.

Meisenheimer, *Ber.*, 1903, **36**, 4177.

Bamberger, Hübner, *ibid.*, 3809.

Ingold, *J. Chem. Soc.*, 1925, **127**, 517.

**4-Nitroso-5-nitro-1-naphthol** (5-Nitro-1:4-naphthoquinone 4-oxime)



$C_{10}H_6O_4N_2$

MW, 218

Yellow needles from AcOH or EtOH. Decomp. at 250–60°. Sol. EtOH, Et<sub>2</sub>O, AcOH, Py. Insol. H<sub>2</sub>O. Sol. alkalis with yellow col. Alk.  $K_3Fe(CN)_6$  → 4:5-dinitro-1-naphthol. Alk.  $KMnO_4$  → 3-nitrophthalic acid.  $SnCl_2 + HCl$  in cold → 4:5-diamino-1-naphthol.  $Sn + HCl$  in hot → 5:8-dihydroxy-1-naphthylamine.

*Acetyl*: cryst. from AcOH. M.p. 136°.

*Benzoyl*: yellow needles. M.p. 210°.

Badische, D.R.P., 90,414.

Friedländer, *Ber.*, 1899, **32**, 3528.

Friedländer, v. Scherzer, *Chem. Zentr.*, 1900, **I**, 410.

Graebe, Oeser, *Ann.*, 1904, **335**, 145.

**4-Nitroso-7-nitro-1-naphthol** (7-Nitro-1:4-naphthoquinone 4-oxime).

Yellowish-brown needles from EtOH.Aq. Decomp. above 200°. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with yellow col. Sol. alkalis with reddish-brown col.  $KMnO_4$  → 4-nitrophthalic acid.

Graebe, *Ann.*, 1904, **335**, 144.

**4-Nitroso-8-nitro-1-naphthol** (8-Nitro-1:4-naphthoquinone 4-oxime).

Yellow cryst. from AcOH. Decomp. at 235–40°. Sol. AcOH. Insol. H<sub>2</sub>O. Sol. conc. H<sub>2</sub>SO<sub>4</sub> and alkalis with yellow col. Alk.  $K_3Fe(CN)_6$  → 4:8-dinitro-1-naphthol.

*Benzoyl*: needles from xylene. M.p. 194°.

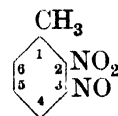
Badische, D.R.P., 91,391.

Friedländer, *Ber.*, 1899, **32**, 3528.

Friedländer, v. Scherzer, *Chem. Zentr.*, 1900, **I**, 411.

Graebe, Oeser, *Ann.*, 1904, **335**, 153.

### 3-Nitroso-o-nitrotoluene



$C_7H_6O_3N_2$

MW, 166

Yellow leaflets from CHCl<sub>3</sub>. M.p. 92–3° decomp. → a green col. Spar. sol. usual org. solvents.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1173.

### 4-Nitroso-o-nitrotoluene.

Colourless needles from EtOH.Aq. M.p. 87° → a green col.

Brand, Zöller, *Ber.*, 1907, **40**, 3333.

**5-Nitroso-*o*-nitrotoluene.**

Cryst. from EtOH. M.p. 113°. Fuming  $\text{HNO}_3 \rightarrow 2:5$ -dinitrotoluene.

Kenner, Parkin, *J. Chem. Soc.*, 1920, **117**, 859.

**6-Nitroso-*o*-nitrotoluene.**

Colourless needles from  $\text{C}_6\text{H}_6$ . M.p. 117°  $\rightarrow$  a green col.

Brand, Zöller, *Ber.*, 1907, **40**, 3331.

**2-Nitroso-*m*-nitrotoluene.**

Yellow leaflets from EtOH. M.p. 126-7° decomp.  $\rightarrow$  green col. Spar. sol. with green col. in org. solvents. Fuming  $\text{HNO}_3 \rightarrow 2:3$ -dinitrotoluene.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1172.

**4-Nitroso-*m*-nitrotoluene.**

Pale yellow needles from EtOH. M.p. 145-145.5°. Sol. hot  $\text{CHCl}_3$ , hot  $\text{Me}_2\text{CO}$ , hot  $\text{C}_6\text{H}_6$ . Mod. sol. hot EtOH, hot ligroin. Spar. sol.  $\text{Et}_2\text{O}$ . Sols. are green in col. Fuming  $\text{HNO}_3 \rightarrow 3:4$ -dinitrotoluene.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1167.

Bamberger, Hübner, *Ber.*, 1903, **36**, 3821.

**6-Nitroso-*m*-nitrotoluene.**

Leaflets from EtOH. M.p. 143-4° decomp. Spar. sol. with green col. in org. solvents. Volatile in steam.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1174.

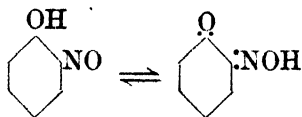
**3-Nitroso-*p*-nitrotoluene.**

Yellow leaflets from  $\text{CHCl}_3$ . M.p. 141°  $\rightarrow$  a green col. Sol. with green col. in most org. solvents.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1169.

***p*-Nitrosophenetole.**

See under *p*-Nitrosophenol.

***o*-Nitrosophenol** (*o*-Benzoquinone monoxime)

$\text{C}_6\text{H}_5\text{O}_2\text{N}$

MW, 123

Pale greenish-yellow needles from pet. ether. Gives deep green sols with org. solvents.

*Cu salt*: deep red needles from EtOH.

*Me ether*: *o*-nitrosoanisole.  $\text{C}_7\text{H}_7\text{O}_2\text{N}$ . MW, 137. Leaflets from  $\text{Me}_2\text{CO}$ -pet. ether. M.p. 103° decomp. Sol. EtOH, hot  $\text{H}_2\text{O}$ , AcOH,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Less sol.  $\text{Et}_2\text{O}$ . Spar. sol. pet.

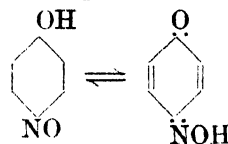
ether.  $\text{KHSO}_4$ . Aq.  $\rightarrow$  *o*-nitrophenol. Volatile in steam.

Baudisch, Karzew, *Ber.*, 1912, **45**, 1169.

Baudisch, Rothschild, *Ber.*, 1915, **48**, 1661.

Baudisch, *Ber.*, 1918, **51**, 1058.

Baeyer, Knorr, *Ber.*, 1902, **35**, 3036.

***p*-Nitrosophenol** (*p*-Benzoquinone monoxime)

$\text{C}_6\text{H}_5\text{O}_2\text{N}$

MW, 123

Pale yellow needles. Turns brown at 124°. Decomp. at 144°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ . Mod. sol.  $\text{H}_2\text{O}$ . Sol. alkalis to brown sols turning green on dilution.  $k = 3.3 \times 10^{-7}$  at 25°. Heat of comb.  $\text{C}_v$  715.5 Cal.

*Me ether*: *p*-nitrosoanisole.  $\text{C}_7\text{H}_7\text{O}_2\text{N}$ . MW, 137. Bluish-green prisms from  $\text{Et}_2\text{O}$ . M.p. 32-4° (23°). Sol. most org. solvents. Spar. sol. ligroin. Insol.  $\text{H}_2\text{O}$ . Easily volatile in steam. Dil.  $\text{H}_2\text{SO}_4 \rightarrow$  *p*-nitrosophenol.

*Et ether*: *p*-nitrosophenetole.  $\text{C}_8\text{H}_9\text{O}_2\text{N}$ . MW, 151. Bluish-green prisms from pet. ether. M.p. 33-4°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether. Insol.  $\text{H}_2\text{O}$ .

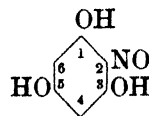
Bridge, *Ann.*, 1893, **277**, 85.

Rising, *Ber.*, 1904, **37**, 44, 46.

Gulinov, *Chem. Zentr.*, 1930, **I**, 972.

Tseng, Hu, *J. Chinese Chem. Soc.*, 1933, **1**, 183.

See also last reference above.

**Nitrosophloroglucinol** (2-Nitroso-1:3:5-trihydroxybenzene, 3:5-dihydroxy-*o*-benzoquinone 2-oxime)

$\text{C}_6\text{H}_5\text{O}_4\text{N}$

MW, 155

1-*Me ether*:  $\text{C}_7\text{H}_7\text{O}_4\text{N}$ . MW, 169. Dark red needles from 50% EtOH. Sol. EtOH. Insol.  $\text{H}_2\text{O}$ . Explodes on heating.

1:3-Di-*Me ether*:  $\text{C}_8\text{H}_9\text{O}_4\text{N}$ . MW, 183. Yellow needles. M.p. 222°. Sol. boiling  $\text{H}_2\text{O}$ . Mod. sol. EtOH,  $\text{Et}_2\text{O}$ .

1:5-Di-*Me ether*: red leaflets. M.p. 175-6°. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. boiling  $\text{H}_2\text{O}$ , AcOEt.

1:3-Di-*Et ether*:  $\text{C}_{10}\text{H}_{13}\text{O}_4\text{N}$ . MW, 211. Pale yellow needles from EtOH or hot  $\text{H}_2\text{O}$ .

M.p. 192–5° decomp. Very sol. EtOH. Insol. Et<sub>2</sub>O.

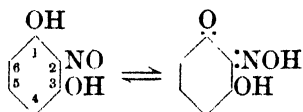
1:5-Di-Et ether: red leaflets from Et<sub>2</sub>O. M.p. 117°. Sol. EtOH, AcOEt, AcOH. Insol. cold H<sub>2</sub>O. Sol. dil. alkalis with brownish-yellow col. Sublimes.

Weidel, Pollak, *Monatsh.*, 1897, **18**, 358; 1900, **21**, 29.

Moldauer, *Monatsh.*, 1896, **17**, 464.

Pollak, Gans, *Monatsh.*, 1902, **23**, 949.

**2-Nitrosoresorcinol** (2-Nitroso-1:3-dihydroxybenzene, 3-hydroxy-o-benzoquinone 2-oxime)



C<sub>6</sub>H<sub>5</sub>O<sub>3</sub>N

MW, 139

3-Et ether: C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 167. Yellow needles. M.p. 102°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Kietabl, *Monatsh.*, 1898, **19**, 544.

Fabre, *Ann. chim.*, 1922, **18**, 49

**4-Nitrosoresorcinol** (4-Nitroso-1:3-dihydroxybenzene, 4-hydroxy-o-benzoquinone 1-oxime, 2-hydroxy-p-benzoquinone 1-oxime).

Yellow needles + H<sub>2</sub>O from H<sub>2</sub>O, brown needles from CHCl<sub>3</sub>. Decomp. at 150°. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>. Less sol. H<sub>2</sub>O, Et<sub>2</sub>O.

1-Me ether: C<sub>7</sub>H<sub>7</sub>O<sub>3</sub>N. MW, 153. (i) *Stable form*: yellowish-brown prisms. M.p. 158–9°. Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, AcOEt, CS<sub>2</sub>. Heat in C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub> or toluene → *labile form*. (ii) *Labile form*: green dichroic plates. At 140° → *stable form*.

3-Me ether: yellow leaflets from AcOH. Decomp. at 160–70°. Sol. EtOH, AcOH. Spar. sol. AcOEt. Insol. cold Et<sub>2</sub>O, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin.

Di-Me ether: C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 167. Pale yellow prisms from ligroin. M.p. 115–17°. Sol. EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold ligroin.

1-Et ether: C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 167. (i) *Stable form*: golden-yellow leaflets. M.p. 146–7°. Sol. warm EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. boiling H<sub>2</sub>O. (ii) *Labile form*: green dichroic cryst. At 130–40° → *stable form*. Benzoyl: yellow cryst. from EtOH. M.p. 155°.

3-Et ether: yellow leaflets from H<sub>2</sub>O, needles from EtOH. Darkens at 160–70°. Sol. hot H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Di-Et ether: C<sub>10</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 195. Pale orange cryst. from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 89–5–

91–5°. Sol. EtOH, AcOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Mod. sol. hot ligroin.

Weselsky, Benedikt, *Monatsh.*, 1880, **1**, 896.

Kietabl, *Monatsh.*, 1898, **19**, 548.

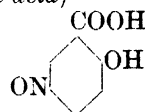
Henrich, *Ber.*, 1902, **35**, 4191; *J. prakt. Chem.*, 1904, **70**, 317.

Henrich, Eisenach, *J. prakt. Chem.*, 1904, **70**, 337.

Henrich, Rhodius, *Ber.*, 1902, **35**, 1478.

Fabre, *Ann. chim.*, 1922, **18**, 49.

**5-Nitrososalicylic Acid** (p-Benzoquinone-oxime 2-carboxylic acid)



C<sub>7</sub>H<sub>5</sub>O<sub>4</sub>N

MW, 167

Green needles from C<sub>6</sub>H<sub>6</sub>. M.p. 162–3° decomp. (rapid heat.). Explodes at 150° with slow heating. Sol. hot H<sub>2</sub>O, Et<sub>2</sub>O and most other org. solvents. Gives green sols.

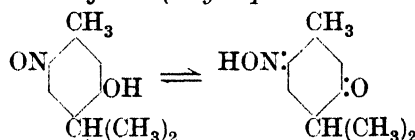
Me ester: C<sub>8</sub>H<sub>7</sub>O<sub>4</sub>N. MW, 181. Blue cubes from H<sub>2</sub>O or pet. ether. M.p. 89–90°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Sublimes. Volatile in steam. Alc. FeCl<sub>3</sub> → deep red col.

Et ester: C<sub>9</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 195. Blue needles from pet. ether. M.p. 47–8°.

Houben, Brassert, Ettinger, Kellner, *Ber.*, 1909, **42**, 2757.

Houben, Schreider, *Ber.*, 1920, **53**, 2356.

**6-Nitrosothymol** (Thymoquinone 1-oxime)



C<sub>10</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 179

Pale yellow needles from CHCl<sub>3</sub>. M.p. 160–4° (160–2°, rapid heat.). Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. boiling H<sub>2</sub>O. Sol. alkalis with yellowish-red col. Heat of comb. C<sub>p</sub> 1334–3 Cal. Alk. K<sub>3</sub>Fe(CN)<sub>6</sub> → 4-nitrothymol.

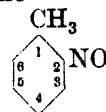
Semicarbazone: m.p. 221–2°.

Schiff, *Ber.*, 1875, **8**, 1500.

Klages, *Ber.*, 1899, **32**, 1518.

Kremers, Wakeman, Hixon, *Organic Syntheses*, Collective Vol. I, 498.

**o-Nitrosotoluene**



C<sub>7</sub>H<sub>7</sub>ON

MW, 121



Needles or prisms. M.p. 72.5°. Very sol.  $\text{CHCl}_3$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Volatile in steam.

Bamberger, Rising, *Ann.*, 1901, **316**, 279.

Bamberger, Tschirner, *Ber.*, 1899, **32**, 1677.

**m-Nitrosotoluene.**

Needles. M.p. 53–53.5°. Volatile in steam.

Bamberger, Rising, *Ann.*, 1901, **316**, 284.

Bamberger, *Ber.*, 1895, **28**, 248.

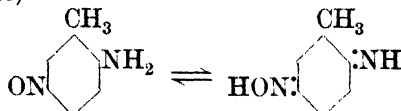
**p-Nitrosotoluene.**

Needles from ligroin. M.p. 48.5°. Sol. hot MeOH,  $\text{C}_6\text{H}_6$ . Less sol. hot ligroin. Spar. sol.  $\text{H}_2\text{O}$ . Volatile in steam.

See previous references and also

Wieland, Roseau, *Ber.*, 1915, **48**, 1119, (Note 1).

**5-Nitroso-o-toluidine** (p-Toluquinoneimine 4-oxime)



$\text{C}_7\text{H}_9\text{ON}_2$  MW, 136

Green needles from  $\text{C}_6\text{H}_6$ . M.p. 115–16° decomp. Sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{H}_2\text{O}$ . Spar. sol. ligroin. Hot NaOH  $\rightarrow$  5-nitroso-o-cresol.

N-Me:  $\text{C}_8\text{H}_{10}\text{ON}_2$ . MW, 150. Green leaflets from  $\text{C}_6\text{H}_6$ . M.p. 151°. Hot dil. NaOH  $\rightarrow$  5-nitroso-o-cresol.

N-Et:  $\text{C}_9\text{H}_{12}\text{ON}_2$ . MW, 164. Green leaflets with blue reflex. M.p. 140°.

N-Butyl:  $\text{C}_{11}\text{H}_{16}\text{ON}_2$ . MW, 192. Blue needles from  $\text{Et}_2\text{O}$ -pet. ether. M.p. 50°. Sol. MeOH,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{CS}_2$ . Spar. sol.  $\text{Et}_2\text{O}$ . Insol. pet. ether. B,HCl: greenish-yellow powder. Decomp. at 136°.

N-Acetyl: green needles from EtOH. M.p. 135–6°. Spar. sol.  $\text{Et}_2\text{O}$ .

Mehne, *Ber.*, 1888, **21**, 731.

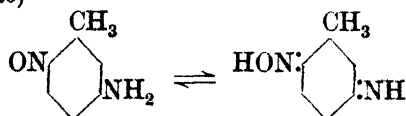
Gnehm, Schröter, *J. prakt. Chem.*, 1906, **73**, 2.

Fischer, Diepolder, *Ann.*, 1895, **286**, 163.

Cain, *J. Chem. Soc.*, 1909, **95**, 715.

Reilly, Hickinbottom, *J. Chem. Soc.*, 1918, **113**, 982.

**6-Nitroso-m-toluidine** (p-Toluquinoneimine 1-oxime)



$\text{C}_7\text{H}_9\text{ON}_2$  MW, 136

Steel-blue needles from  $\text{C}_6\text{H}_6$ . M.p. 178°. Sol. EtOH,  $\text{Et}_2\text{O}$ . Insol. ligroin.

N-Di-Me:  $\text{C}_9\text{H}_{12}\text{ON}_2$ . MW, 164. Green leaflets from  $\text{Et}_2\text{O}$ . M.p. 92°. Gives intense green sols.

N-Acetyl: green needles from EtOH. M.p. 128–9°. Sol. AcOH. Spar. sol.  $\text{Et}_2\text{O}$ .

Wurster, Riedel, *Ber.*, 1879, **12**, 1797.

Mehne, *Ber.*, 1888, **21**, 730.

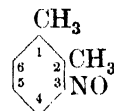
Cain, *J. Chem. Soc.*, 1909, **95**, 715.

I.G., D.R.P., 561,424, (*Chem. Zentr.*, 1933, II, 444).

**4-Nitrosoveratrol.**

See under 5-Nitrosoguaiacol.

**3-Nitroso-o-xylene**



$\text{C}_8\text{H}_9\text{ON}$  MW, 135

Needles. M.p. 91–91.5°. Sol. warm EtOH,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. sol. pet. ether. Volatile in steam. Reduces alc. Fehling's on heating.

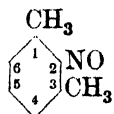
Bamberger, Rising, *Ann.*, 1901, **316**, 287.

**4-Nitroso-o-xylene.**

Pale bluish-green needles from EtOH. M.p. 44–5°. Very sol.  $\text{Me}_2\text{CO}$ , warm EtOH. Mod. sol. pet. ether. Reduces hot Fehling's in presence of EtOH.

See previous reference.

**2-Nitroso-m-xylene**



$\text{C}_8\text{H}_9\text{ON}$  MW, 135

Needles. M.p. 144–5° (141.5°). Very sol. warm EtOH,  $\text{Me}_2\text{CO}$ . Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ , ligroin. Does not reduce Fehling's.

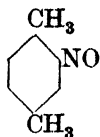
v. Pechmann, Nold, *Ber.*, 1898, **31**, 560.

Bamberger, Rising, *Ann.*, 1901, **316**, 309.

**4-Nitroso-m-xylene.**

Prisms from EtOH. M.p. 41.5°. Very sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Sol. pet. ether, ligroin. Volatile in steam.

See previous references.

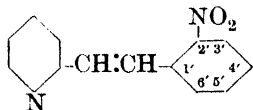
2-Nitroso-*p*-xylene $C_8H_9ON$ 

MW, 135

Needles from EtOH. M.p. 101.5°. Spar. sol. Et<sub>2</sub>O, pet. ether.

Bamberger, Rising, *Ann.*, 1901, 316, 289.

**2'-Nitro- $\alpha$ -stilbazole** (2-*o*-Nitrostyrylpyridine)

 $C_{13}H_{10}O_2N_2$ 

MW, 226

*Cis*:

Green needles from CS<sub>2</sub>. M.p. 95°.

*Trans*:

Yellow prismatic needles from EtOH. M.p. 101° (95-6°). Sol. CHCl<sub>3</sub>.

*B, HCl*: needles from HCl. M.p. 216° (206-12°) decomp.

*B, HNO<sub>3</sub>*: needles. M.p. 148°.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: reddish-yellow cryst. M.p. 220-4° decomp.

*Picrate*: yellow needles from Me<sub>2</sub>CO. M.p. 220°.

Rath, Lehmann, *Ber.*, 1925, 58, 343.

Shaw, Wagstaff, *J. Chem. Soc.*, 1933, 78.

**3'-Nitro- $\alpha$ -stilbazole** (3-*m*-Nitrostyrylpyridine).

Pale yellow needles or leaflets from EtOH. M.p. 129° (120°). Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>. Spar. sol. hot H<sub>2</sub>O.

*B, HCl*: yellow needles from H<sub>2</sub>O. M.p. 230°.

*B, H<sub>2</sub>AuCl<sub>4</sub>*: pale yellow cryst. powder. M.p. 187°.

*B, HCl, HgCl<sub>2</sub>*: yellowish needles. M.p. 211° decomp.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: yellow needles. M.p. 240° decomp.

Feist, *Ber.*, 1901, 34, 465.

Shaw, Wagstaff, *J. Chem. Soc.*, 1933, 78.

**4'-Nitro- $\alpha$ -stilbazole** (2-*p*-Nitrostyrylpyridine).

Yellow prisms from pet. ether. M.p. 136° (124°). *D*<sub>4</sub><sup>20</sup> 1.319. Darkens in air.

*B, HNO<sub>3</sub>*: m.p. 155° decomp.

*B<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>*: m.p. 274°.

*B, H<sub>2</sub>AuCl<sub>4</sub>*: greyish-yellow powder. M.p. 205° decomp.

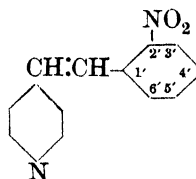
Dict. of Org. Comp.—III.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: grey powder. M.p. 206-7° decomp.

*Picrate*: yellow needles from EtOH. M.p. 272°.

See previous references.

**2'-Nitro- $\gamma$ -stilbazole** (4-*o*-Nitrostyrylpyridine)

 $C_{13}H_{10}O_2N_2$ 

MW, 226

Cryst. from EtOH. M.p. 98-100°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*B, HCl*: m.p. 191-2°.

*B, HNO<sub>3</sub>*: m.p. 95°.

*B, H<sub>2</sub>SO<sub>4</sub>*: m.p. 110°.

*B, H<sub>2</sub>AuCl<sub>4</sub>*: m.p. 215°.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 206°.

*B, HCl, HgCl<sub>2</sub>*: m.p. 175-6°.

*Picrate*: m.p. 198°.

Löwensohn, *Ber.*, 1907, 40, 4860.

**3'-Nitro- $\gamma$ -stilbazole** (4-*m*-Nitrostyrylpyridine).

Brown needles from EtOH. M.p. 138°.

*B, HCl*: powder from EtOH-Et<sub>2</sub>O. M.p. 221-2°.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: yellowish-red leaflets.

*Picrate*: yellowish-green cryst. Explosive.

Friedländer, *Ber.*, 1905, 38, 2838.

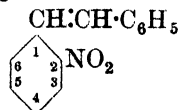
**4'-Nitro- $\gamma$ -stilbazole** (4-*p*-Nitrostyrylpyridine).

Yellow needles from EtOH.Aq. M.p. 118-19°. Sol. EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Sublimes in vacuo.

*B, HCl*: cryst. from alc. HCl. M.p. 257-8°.

Baumert, *Ber.*, 1906, 39, 2971.

## 2-Nitrostilbene

 $C_{14}H_{11}O_2N$ 

MW, 225

Exists in two forms.

(i) Pale yellow needles from EtOH. M.p. 78° (72-3°). Sol. usual solvents. Sol. 100 parts pet. ether at 18°. Irradiation by Hg vapour lamp  $\rightarrow$  second form.

(ii) Yellow cryst. from pet. ether. M.p. about 42°. Sol. 50 parts pet. ether at 18°.

Stoermer, Prigge, *Ann.*, 1915, **409**, 34.

Pfeiffer, *Ber.*, 1915, **48**, 1052.

#### 4-Nitrostilbene.

Exists in two forms.

(i) Pale yellow needles from EtOH. M.p. 155°. Sol. Et<sub>2</sub>O, AcOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, warm EtOH. Spar. sol. ligroin.

(ii) Brownish-yellow plates from EtOH. M.p. 65°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>. C<sub>6</sub>H<sub>6</sub> + 1 in sunlight → first form.

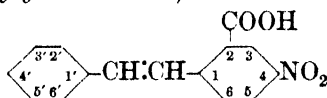
Stoermer, Oehlert, *Ber.*, 1922, **55**, 1239.

Pfeiffer, Sergiewskaja, *Ber.*, 1911, **44**, 1109.

#### Nitrostilbene- $\alpha$ -carboxylic Acid

See  $\alpha$ -Nitrophenylcinnamic Acid.

#### 4-Nitrostilbene-2-carboxylic Acid (5-Nitro-*o*-styrylbenzoic acid)



C<sub>15</sub>H<sub>11</sub>O<sub>4</sub>N

MW, 269

Yellow needles from 50% EtOH. M.p. 206°. Sol. Me<sub>2</sub>CO. Mod. sol. MeOH, EtOH, Et<sub>2</sub>O, AcOH, hot C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

*Nitrile*: 4-nitro-2-cyanostilbene. C<sub>15</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>. MW, 250. Yellow needles from AcOH. M.p. 142°. Sol. boiling AcOH. Mod. sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O.

Ullmann, Gschwind, *Ber.*, 1908, **41**, 2296.

Pfeiffer, Matton, *Ber.*, 1911, **44**, 1119.

#### 4-Nitrostilbene-3-carboxylic Acid (6-Nitro-*m*-styrylbenzoic acid).

Yellow leaflets from AcOH. M.p. 203°. Mod. sol. EtOH, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin.

*Nitrile*: 4-nitro-3-cyanostilbene. Yellow needles from AcOH. M.p. 187°. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. hot AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin.

Pfeiffer, *Ber.*, 1918, **51**, 560.

#### 2-Nitrostilbene-4-carboxylic Acid (3-Nitro-*p*-styrylbenzoic acid).

Exists in two forms.

(i) *Labile form*.

Pale yellow cryst. from EtOH. M.p. 158°. Sunlight on C<sub>6</sub>H<sub>6</sub> sol. + I → stable form.

*Me ester*: C<sub>16</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 283. Prisms from EtOH. M.p. 91°. Sunlight → stable form.

(ii) *Stable form*.

Yellow needles from AcOH. M.p. 236°. Sol. boiling EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Prolonged irradiation → labile form.

*Me ester*: needles from MeOH or EtOH. M.p. 122°. Mod. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Spar. sol. ligroin.

*Et ester*: C<sub>17</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 297. Pale yellow needles from EtOH. M.p. 124°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Spar. sol. ligroin.

*Nitrile*: 2-nitro-4-cyanostilbene. Yellow needles or leaflets from AcOH. M.p. 170°. Sol. AcOH. Spar. sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Quinine salt*: pale yellow cryst. from CS<sub>2</sub>. M.p. 185°.

*Cinchonine salt*: yellow prisms from EtOH. M.p. 156-7°.

*Strychnine salt*: yellow cryst. from EtOH. M.p. 219°.

*Brucine salt*: yellow needles from EtOH. M.p. 165-8°.

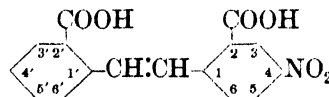
Ullmann, Gschwind, *Ber.*, 1908, **41**, 2295.

Pfeiffer, Matton, *Ber.*, 1911, **44**, 1123.

Stoermer, Oehlert, *Ber.*, 1922, **55**, 1241.

Pfeiffer, du Plessis, Richarz, Stallmann, *J. prakt. Chem.*, 1930, **127**, 172.

#### 4-Nitrostilbene-2 : 2'-dicarboxylic Acid



C<sub>16</sub>H<sub>11</sub>O<sub>6</sub>N

MW, 313

Yellow needles from *m*-cresol. M.p. about 248°. Very sol. Py. Sol. Me<sub>2</sub>CO, warm EtOH, AcOH. Spar. sol. hot C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

*2-Nitrile*: C<sub>16</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 294. Pale yellow needles from AcOH. M.p. 182°. Sol. Py. Mod. sol. Me<sub>2</sub>CO, hot EtOH. Spar. sol. Et<sub>2</sub>O, boiling C<sub>6</sub>H<sub>6</sub>. Insol. pet. ether. *Et ester*: C<sub>18</sub>H<sub>14</sub>O<sub>4</sub>N<sub>2</sub>. MW, 322. Yellow leaflets or needles from EtOH. M.p. 133°. Sol. AcOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH.

Pfeiffer, Matton, *Ber.*, 1911, **44**, 1118.

#### 2-Nitrostilbene-2' : 4-dicarboxylic Acid.

Pale yellow needles from *m*-cresol. M.p. 257°. Very sol. Py, Me<sub>2</sub>CO. Sol. EtOH, AcOH. Spar. sol. hot C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

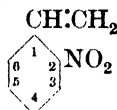
*Di-Me ester*: C<sub>18</sub>H<sub>15</sub>O<sub>6</sub>N. MW, 341. Pale yellow needles from EtOH. M.p. 138°. Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH. Insol. ligroin.

*4-Nitrile*: C<sub>16</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>. MW, 294. Yellow needles from *m*-cresol. M.p. 227°. Sol. Py,

EtOH, AcOH, Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O, warm C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

See previous reference.

## o-Nitrostyrene



C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>N MW, 149

Oil. F.p. 12–13.5°. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with blue col.

Einhorn, *Ber.*, 1883, **16**, 2213.

## m-Nitrostyrene.

Yellow oil. F.p. – 5°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH, ligroin.

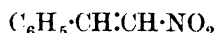
Prausnitz, *Ber.*, 1884, **17**, 597.

## p-Nitrostyrene.

Prisms from ligroin. M.p. 29°. Sol. warm EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Volatile in steam. Decomp. on dist.

Basler, *Ber.*, 1883, **16**, 3003.

## ω-Nitrostyrene



C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>N MW, 149

Yellowish prisms from EtOH or pet. ether. M.p. 58°. B.p. 250–60° decomp. Very sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. ligroin. Spar. sol. hot H<sub>2</sub>O. Volatile in steam. Heat with CaCl<sub>2</sub> → chloropierin. In sunlight → dimeric form, m.p. 180–7° decomp.

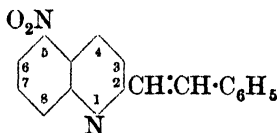
Thiele, Haackel, *Ann.*, 1902, **325**, 7.

Worrall, *Organic Syntheses*, Collective Vol. I, 405.

## Nitrostyrylpyridine.

See Nitrostilbazole.

## 5-Nitro-2-styrylquinoline (5-Nitrobenzylidenequinoline)



C<sub>17</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub> MW, 276

Pale yellow needles from EtOH.Aq. M.p. 127°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, Me<sub>2</sub>CO, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

B, HCl: golden-yellow needles. M.p. 213°. Sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O.

B, H<sub>2</sub>SO<sub>4</sub>: golden-yellow needles. M.p. 237°. Sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O.

B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>: golden-yellow needles. Decomp. above 199°.

B<sub>2</sub>, 2HCl, HgCl<sub>2</sub>: yellow needles. M.p. about 249–50°.

Picrate: yellow needles. M.p. 236°. Spar. sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O.

Schmidt, *Ber.*, 1905, **38**, 3718.

## 6-Nitro-2-styrylquinoline (6-Nitrobenzylidenequinoline).

Pale yellow needles from EtOH. M.p. 192°. Sol. CHCl<sub>3</sub>, Me<sub>2</sub>CO, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O.

B, HCl: pale yellow needles. M.p. 205°. Sol. EtOH.

B, H<sub>2</sub>SO<sub>4</sub>: pale yellow needles. M.p. 218°. Sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O.

B<sub>2</sub>, HCl, HgCl<sub>2</sub>: deep yellow needles. M.p. about 245°. Very spar. sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O.

See previous reference.

## 8-Nitro-2-styrylquinoline (8-Nitrobenzylidenequinoline).

Pale yellow needles from EtOH.Aq. M.p. about 142°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, Me<sub>2</sub>CO.

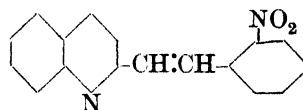
B, HCl: reddish-yellow leaflets. Decomp. about 140°. Sol. EtOH.

B, H<sub>2</sub>SO<sub>4</sub>: orange needles. M.p. 233°.

B<sub>2</sub>, 2HCl, HgCl<sub>2</sub>: orange needles. M.p. 224°.

See previous reference.

## 2-o-Nitrostyrylquinoline (o-Nitrobenzylidenequinoline)



C<sub>17</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub> MW, 276

Yellow cryst. from EtOH. M.p. 103°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

B, HCl: yellow needles from dil. HCl. M.p. 253°.

B<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>: m.p. 238°.

B, HNO<sub>3</sub>: yellow needles from dil. HNO<sub>3</sub>. M.p. 178°.

B, H<sub>2</sub>SO<sub>4</sub>: yellow needles from EtOH. M.p. 241° decomp.

B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>: light brown cryst. M.p. 223°.

Loew, *Ber.*, 1903, **36**, 1666.

## 2-m-Nitrostyrylquinoline (m-Nitrobenzylidenequinoline).

Needles. M.p. 156°.

*Picrate*: m.p. 261° decomp.

Taylor, Woodhouse, *J. Chem. Soc.*, 1926, 2971.

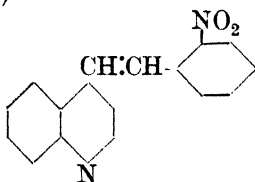
Wallach, Wüsten, *Ber.*, 1883, 16, 2009.

**2-*p*-Nitrostyrylquinoline** (*p*-Nitrobenzyl-*idenequinaldine*).

Needles. M.p. 164–5°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, ligroin, hot EtOH.

Bulach, *Ber.*, 1887, 20, 2047.

**4-*o*-Nitrostyrylquinoline** (*o*-Nitrobenzyl-*idenelepidine*)



C<sub>17</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub> MW, 276

Yellow leaflets from EtOH. M.p. 162°. Sol. CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*B.HCl*: pale yellow leaflets from dil. HCl. M.p. 257–8°.

*B.HNO<sub>3</sub>*: yellow needles. M.p. 178°.

*B.HAuCl<sub>4</sub>*: yellow leaflets from EtOH. M.p. 235°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow cryst. Decomp. at 262°.

*Methiodide*: red cryst. from EtOH. M.p. 237°.

Loew, *Ber.*, 1903, 36, 1669.

**4-*m*-Nitrostyrylquinoline** (*m*-Nitrobenzyl-*idenelepidine*).

Needles from EtOH. M.p. 131–2°. Spar. sol. cold EtOH.

Heyman, Koenigs, *Ber.*, 1888, 21, 1429, 2172.

**4-*p*-Nitrostyrylquinoline** (*p*-Nitrobenzyl-*idenelepidine*).

Yellow needles from EtOH. M.p. 221°.

*B.HCl*: yellow needles from EtOH. M.p. 272°.

*B.HBr*: yellow needles from EtOH. M.p. 297°.

*B.HAuCl<sub>4</sub>*: yellow needles. M.p. 236°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellowish-brown cryst. Does not melt below 300°.

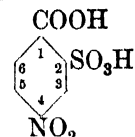
*Picrate*: yellow needles from EtOH. M.p. 287°. Sol. MeOH, EtOH, Me<sub>2</sub>CO.

Loew, *Ber.*, 1903, 36, 1670.

**Nitrosulphanilic Acid.**

See Nitroanilinesulphonic Acid.

**4-Nitro-2-sulphobenzoic Acid** (4-Nitrobenzoic acid 2-sulphonic acid)



C<sub>7</sub>H<sub>5</sub>O<sub>7</sub>NS

MW, 247

Cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 147°. Very sol. EtOH. PCl<sub>5</sub> at 170° and on pouring into H<sub>2</sub>O → 2-chloro-4-nitrobenzoic acid.

1-*Me* ester: C<sub>8</sub>H<sub>7</sub>O<sub>7</sub>NS. MW, 261. Needles from H<sub>2</sub>O. M.p. 270° decomp. Very sol. H<sub>2</sub>O. Spar. sol. MeOH.

1-*Me*-2-*Et* ester: C<sub>10</sub>H<sub>11</sub>O<sub>7</sub>NS. MW, 289. Needles. M.p. 68°.

1-*Et*-2-*Me* ester: prisms from Et<sub>2</sub>O. M.p. 80°.

Di-*Et* ester: C<sub>11</sub>H<sub>13</sub>O<sub>7</sub>NS. MW, 303. Leaflets from EtOH. M.p. 65–6°.

Diphenyl ester: C<sub>19</sub>H<sub>13</sub>O<sub>7</sub>NS. MW, 399. Yellow needles from EtOH. M.p. 119°.

Di-*o*-nitrophenyl ester: C<sub>19</sub>H<sub>11</sub>O<sub>11</sub>N<sub>3</sub>S. MW, 489. Needles from AcOH. M.p. 164°.

Di-*p*-nitrophenyl ester: needles. M.p. 152°.

Di-*p*-tolyl ester: C<sub>21</sub>H<sub>17</sub>O<sub>7</sub>NS. MW, 427. Needles or plates from C<sub>6</sub>H<sub>6</sub>. M.p. 117°.

Di-β-naphthyl ester: C<sub>27</sub>H<sub>17</sub>O<sub>7</sub>NS. MW, 499. Needles from AcOH. M.p. 134°.

1-*Me* ester 2-chloride: C<sub>8</sub>H<sub>6</sub>O<sub>6</sub>NCIS. MW, 279.5. Prisms from MeOH. M.p. 90° (82°).

1-*Et* ester 2-chloride: C<sub>9</sub>H<sub>8</sub>O<sub>6</sub>NCIS. MW, 293.5. Needles from EtOH. M.p. 68°.

1-Phenyl ester 2-chloride: C<sub>13</sub>H<sub>8</sub>O<sub>6</sub>NCIS. MW, 341.5. Needles from AcOH. M.p. 145–7°.

Dichloride: C<sub>7</sub>H<sub>3</sub>O<sub>5</sub>NCl<sub>2</sub>S. MW, 284. Exists in two forms: (i) cryst. from CHCl<sub>3</sub> or Et<sub>2</sub>O. M.p. 98° (94°); (ii) needles or plates from pet. ether. M.p. 56–7°. Very sol. CHCl<sub>3</sub>, Et<sub>2</sub>O.

Nitrile: C<sub>7</sub>H<sub>4</sub>O<sub>5</sub>N<sub>2</sub>S. MW, 228. Prisms + 1H<sub>2</sub>O. M.p. 140–50° (rapid heat.). Very sol. H<sub>2</sub>O.

2-Chloride: C<sub>7</sub>H<sub>3</sub>O<sub>5</sub>N<sub>2</sub>ClS. MW, 246.5. Prisms. M.p. 107–8°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

Dianilide: C<sub>19</sub>H<sub>15</sub>O<sub>5</sub>N<sub>3</sub>S. MW, 397. Needles from EtOH. M.p. 222°.

Kastle, *Am. Chem. J.*, 1889, 11, 179.

Hausser, *Bull. soc. chim.*, 1891, 6, 391.

Henderson, *Am. Chem. J.*, 1901, 25, 1.

Chambers, *Am. Chem. J.*, 1903, 30, 381.

Holmes, *Am. Chem. J.*, 1901, 25, 204.

**5-Nitro-2-sulphobenzoic Acid** (3-Nitrobenzoic acid 6-sulphonic acid).

M.p. (+2H<sub>2</sub>O) 105°, anhyd. 153°. Hydrate sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O. Insol.

$\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether.  $\text{PCl}_5 \longrightarrow$  6-chloro-3-nitrobenzoic acid.

Stubbs, *Am. Chem. J.*, 1913, 50, 195.

Taverne, *Rec. trav. chim.*, 1906, 25, 64.

**5-Nitro-3-sulphobenzoic Acid** (3-Nitrobenzoic acid 5-sulphonic acid).

Cryst. +  $7\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ , m.p.  $96^\circ$ ; needles +  $1\text{H}_2\text{O}$ , m.p.  $70^\circ$ . M.p. anhyd.  $159.5^\circ$  ( $152^\circ$ ).

3-Chloride:  $\text{C}_7\text{H}_4\text{O}_6\text{NClS}$ . MW, 265.5. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $170^\circ$ .

Dichloride:  $\text{C}_7\text{H}_3\text{O}_5\text{NCl}_2\text{S}$ . MW, 284. Prisms. M.p.  $64^\circ$ . Sol.  $\text{C}_6\text{H}_6$ , toluene. Mod. sol.  $\text{CCl}_4$ .

3-Amide:  $\text{C}_7\text{H}_6\text{O}_6\text{N}_2\text{S}$ . MW, 246. Plates. M.p.  $230^\circ$ .

Diamide:  $\text{C}_7\text{H}_7\text{O}_5\text{N}_3\text{S}$ . MW, 245. Needles from  $\text{H}_2\text{O}$ . M.p.  $226^\circ$ .

van Dorssen, *Rec. trav. chim.*, 1910, 29, 382.

Taverne, *Rec. trav. chim.*, 1906, 25, 67.

Shah, Bhatt, *J. Chem. Soc.*, 1933, 1373.

**2-Nitro-4-sulphobenzoic Acid** (2-Nitrobenzoic acid 4-sulphonic acid).

Hygroscopic needles +  $2\frac{1}{2}\text{H}_2\text{O}$ . M.p.  $111^\circ$ .

1-Me ester: cryst. +  $2\text{H}_2\text{O}$ . M.p.  $95-7^\circ$ . Hygroscopic. Sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Prac. insol.  $\text{C}_6\text{H}_6$ .

4-Me ester: m.p.  $140-2^\circ$ . Insol. cold  $\text{H}_2\text{O}$ .

Di-Me ester: cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $86-7^\circ$ . Sol.  $\text{Et}_2\text{O}$ , hot  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH. Insol.  $\text{H}_2\text{O}$ .

Dichloride: m.p.  $160^\circ$ .

4-Amide: m.p.  $192^\circ$ .

Diamide: needles. M.p.  $226^\circ$ .

Wegscheider, Furcht, *Monatsh.*, 1902, 23, 1139.

Beck, D.R.P., 80,165.

Hart, *Am. Chem. J.*, 1879, 1, 352.

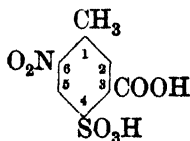
Hirwe, Jambhekar, *J. Indian Chem. Soc.*, 1933, 10, 47.

**3-Nitro-4-sulphobenzoic Acid** (3-Nitrobenzoic acid 4-sulphonic acid).

Prisms +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $130-1^\circ$ , anhyd.  $159^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ .

Hart, *Am. Chem. J.*, 1879, 1, 343.

**6-Nitro-4-sulpho-m-toluic Acid** (6-Nitro-m-toluic acid 4-sulphonic acid)



$\text{C}_8\text{H}_7\text{O}_7\text{NS}$

MW, 261

Pale yellowish needles +  $3\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd.  $94^\circ$ . Sol.  $\text{Me}_2\text{CO}$ , AcOH, AcOEt. Insol.  $\text{Et}_2\text{O}$ ,  $\text{CCl}_4$ , ligroin.

Di-Me ester:  $\text{C}_{10}\text{H}_9\text{O}_7\text{NS}$ . MW, 289. Cryst. from MeOH. M.p.  $94.5^\circ$ . Insol.  $\text{H}_2\text{O}$ .

Diphenyl ester:  $\text{C}_{20}\text{H}_{15}\text{O}_7\text{NS}$ . MW, 413. Cryst. from EtOH. M.p.  $123^\circ$ .

4-Chloride:  $\text{C}_8\text{H}_6\text{O}_6\text{NClS}$ . MW, 279.5. Me ester:  $\text{C}_9\text{H}_8\text{O}_6\text{NClS}$ . MW, 293.5. Plates from ligroin. M.p.  $101^\circ$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Et ester:  $\text{C}_{10}\text{H}_{10}\text{O}_6\text{NClS}$ . MW, 297.5. M.p.  $72^\circ$ .

Dichloride:  $\text{C}_8\text{H}_5\text{O}_5\text{NCl}_2\text{S}$ . MW, 298. Exists in two isomeric forms. (i) Rhombohedra or needles from  $\text{CCl}_4$ . M.p.  $134^\circ$ . (ii) Yellowish needles from  $\text{CCl}_4$ . M.p.  $83^\circ$ . B.p.  $218-20^\circ/21\text{ mm}$ .

Karslake, Bond, *J. Am. Chem. Soc.*, 1909, 31, 408; 1916, 38, 1339.

**4-Nitro-6-sulpho-m-toluic Acid** (4-Nitro-m-toluic acid 6-sulphonic acid).

Needles +  $\text{H}_2\text{O}$ . M.p.  $34-7^\circ$ , anhyd.  $150.7^\circ$ . Very sol.  $\text{H}_2\text{O}$  and most org. solvents.

Di-Me ester: cryst. Decomp. at  $302-5^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Sol. MeOH, EtOH,  $\text{CHCl}_3$ ,  $\text{CCl}_4$ ,  $\text{C}_6\text{H}_6$ , ligroin.

Dichloride: prismatic plates from  $\text{CCl}_4$  or  $\text{CHCl}_3$ . M.p.  $90.2^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{CCl}_4$ ,  $\text{C}_6\text{H}_6$ .

Diamide:  $\text{C}_8\text{H}_9\text{O}_5\text{N}_3\text{S}$ . MW, 259. Prismatic plates from 50% EtOH. M.p.  $273-4^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

Dianilide:  $\text{C}_{20}\text{H}_{17}\text{O}_5\text{N}_3\text{S}$ . MW, 411. Yellow plates from 70% EtOH. M.p.  $244.8^\circ$ . Sol. most org. solvents.

Di-o-toluidide:  $\text{C}_{22}\text{H}_{21}\text{O}_5\text{N}_3\text{S}$ . MW, 439. Greenish-yellow prisms from 70% EtOH. M.p.  $238.7^\circ$ . Sol. most org. solvents. Insol.  $\text{H}_2\text{O}$ .

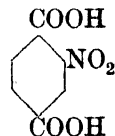
Di-m-toluidide: yellow needles or prisms from EtOH.Aq. M.p.  $208.8^\circ$ .

Di-p-toluidide: needles from EtOH.Aq. M.p.  $241.8^\circ$ .

Karslake, Bond, *J. Am. Chem. Soc.*, 1909, 31, 406.

Karslake, Huston, *ibid.*, 1058.

**2-Nitroterephthalic Acid**



$\text{C}_8\text{H}_5\text{O}_6\text{N}$

MW, 211

Needles from hot  $\text{H}_2\text{O}$ . M.p.  $268^\circ$  ( $262-3^\circ$ ). Sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ .  $k$  (first) =  $1.87 \times 10^{-2}$  at  $25^\circ$ ; (second) =  $2.0 \times 10^{-4}$  at  $25^\circ$ .

### 5-Nitro-1 : 2 : 3 : 4-tetrahydronaphthalene

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1-*Me ester*:  $C_9H_7O_6N$ . MW, 225. Cryst. from  $C_6H_6$  or  $H_2O$ . M.p. 175-6°.  $k = 7.7 \times 10^{-4}$  at 25°. 1-*Menthyl ester*: cryst. from  $C_6H_6$ . M.p. 72-4°.  $D_4^{20}$  1.133.  $[\alpha]_D^{20} - 68.31^\circ$ .

4-*Me ester*: needles from  $C_6H_6$ . M.p. 133.5-135°. Mod. sol.  $H_2O$ .  $k = 1.9 \times 10^{-2}$  at 25°. 1-*Menthyl ester*: needles. M.p. 78-9°.  $[\alpha]_D^{18} - 141.7^\circ$  in  $C_6H_6$ .

Di-*Me ester*:  $C_{10}H_9O_6N$ . MW, 239. Plates from EtOH. M.p. 76°. Sol.  $C_6H_6$ . Spar. sol. pet. ether. Volatile in steam.

1-*Et ester*:  $C_{10}H_9O_6N$ . MW, 239. Cryst. from  $C_6H_6$ . M.p. 146-8°. 1-*Menthyl ester*: cryst. from  $C_6H_6$ . M.p. 48°.  $D_4^{20}$  1.127.  $[\alpha]_D^{20} - 65.55^\circ$ .

4-*Et ester*: 1-*menthyl ester*, liq.  $D_4^{20}$  1.110.  $[\alpha]_D^{20} - 91.58^\circ$ ,  $[\alpha]_D^{18} - 130.5^\circ$  in  $C_6H_6$ .

Di-*Et ester*:  $C_{12}H_{13}O_6N$ . MW, 267. Needles from EtOH.Aq. M.p. 57°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Mod. sol.  $H_2O$ .

1-*Propyl ester*:  $C_{11}H_{11}O_6N$ . MW, 253. Cryst. from  $C_6H_6$ . M.p. 135-7°. 1-*Menthyl ester*: b.p. 192°/6 mm.  $D_4^{20}$  1.130.  $[\alpha]_D^{20} - 62.25^\circ$ .

4-*Propyl ester*: 1-*menthyl ester*, thick liq.  $D_4^{20}$  1.110.  $[\alpha]_D^{20} - 92.74^\circ$ ,  $[\alpha]_D^{18} - 129.7^\circ$  in  $C_6H_6$ .

Dipropyl ester:  $C_{14}H_{17}O_6N$ . MW, 295. Oil. B.p. 228-30°/18 mm.

1-*Butyl ester*:  $C_{12}H_{13}O_6N$ . MW, 267. Cryst. from  $C_6H_6$ . M.p. 132-4°. 1-*Menthyl ester*: b.p. 215°/6 mm.  $D_4^{20}$  1.136.  $[\alpha]_D^{20} - 60.23^\circ$ .

4-*Butyl ester*: 1-*menthyl ester*, thick liq.  $D_4^{20}$  1.099.  $[\alpha]_D^{20} - 90.51^\circ$ ,  $[\alpha]_D^{18} - 124.7^\circ$  in  $C_6H_6$ .

1-1-*Menthyl ester*: cryst. from EtOH.Aq. M.p. 75°.

Di-1-*Menthyl ester*: needles from EtOH. M.p. 88°.  $[\alpha]_D^{18} - 160.5^\circ$  in  $C_6H_6$ .

Dichloride:  $C_8H_3O_4NCl_2$ . MW, 248. B.p. 174°/8 mm.

Wegscheider, *Monatsh.*, 1900, 21, 622; 1902, 23, 407.

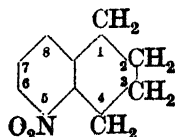
Wegscheider, Gehringer, *Monatsh.*, 1908, 29, 529.

Cohen, de Pennington, *J. Chem. Soc.*, 1918, 113, 64.

Kauffmann, Weissel, *Ann.*, 1912, 393, 10.

Soderman, Johnson, *J. Am. Chem. Soc.*, 1925, 47, 1393.

### 5-Nitro-1 : 2 : 3 : 4-tetrahydronaphthalene (5-Nitrotetralin)



$C_{10}H_{11}O_2N$

MW, 177

### 4-Nitro-5 : 6 : 7 : 8-tetrahydro-2-naphthol

Cryst. from MeOH. M.p. 34°. B.p. 157°/13 mm.  $D_4^{20}$  1.1757.

Schroeter, *Ann.*, 1922, 426, 39.

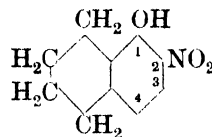
Tetralingesellschaft, D.R.P., 299,014, (*Chem. Zentr.*, 1919, IV, 374).

### 6-Nitro-1 : 2 : 3 : 4-tetrahydronaphthalene (6-Nitrotetralin).

Needles from EtOH or MeOH. M.p. 31.4°. B.p. 169°/13 mm.  $D_4^{20}$  1.1762.

See previous references.

### 2-Nitro-5 : 6 : 7 : 8-tetrahydro-1-naphthol (2-Nitro-ar-tetrahydro- $\alpha$ -naphthol)



$C_{10}H_{11}O_3N$

MW, 193

Yellow needles from  $Et_2O$ . M.p. 56°. Sol. most org. solvents. Spar. sol.  $H_2O$ . Volatile in steam. Sweet odour.

Na salt: orange plates or needles from  $H_2O$ .

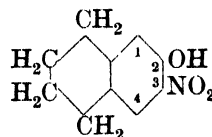
Green, Rowe, *J. Chem. Soc.*, 1918, 113, 968.

### 4-Nitro-5 : 6 : 7 : 8-tetrahydro-1-naphthol (4-Nitro-ar-tetrahydro- $\alpha$ -naphthol).

Needles from toluene. M.p. 123°. Non-volatile in steam. Gives yellowish-brown salts.

Rowe, Levin, *J. Chem. Soc.*, 1927, 531.

### 3-Nitro-5 : 6 : 7 : 8-tetrahydro-2-naphthol (3-Nitro-ar-tetrahydro- $\beta$ -naphthol)



$C_{10}H_{11}O_3N$

MW, 193

Golden-yellow plates from EtOH or  $CCl_4$ , needles from AcOH.Aq. M.p. 88-9°. Spar. sol.  $H_2O$ .

Acetyl: pale yellow cryst. M.p. 100°.

Thoms, Kross, *Chem. Zentr.*, 1927, I, 3000.

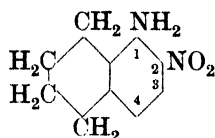
### 4-Nitro-5 : 6 : 7 : 8-tetrahydro-2-naphthol (4-Nitro-ar-tetrahydro- $\beta$ -naphthol).

Amorphous.

Schroeter et al., *Ann.*, 1922, 426, 70.

**2-Nitro-5 : 6 : 7 : 8-tetrahydro-1-naphthylamine**

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**2-Nitro-5 : 6 : 7 : 8-tetrahydro-1-naphthylamine** (2-Nitro-ar-tetrahydro- $\alpha$ -naphthylamine) $C_{10}H_{12}O_2N_2$ 

MW, 192

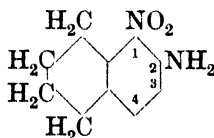
Orange needles from EtOH. M.p. 87–8°.

N-Acetyl: needles from EtOH. M.p. 184–5°.  
Sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. CCl<sub>4</sub>.Schroeter *et al.*, *Ann.*, 1922, 426, 62.**3-Nitro-5 : 6 : 7 : 8-tetrahydro-1-naphthylamine** (3-Nitro-ar-tetrahydro- $\alpha$ -naphthylamine).

Yellow leaflets from EtOH. M.p. 78°.

Acetyl: needles from 60% AcOH. M.p. 194–5°.

See previous reference.

**4-Nitro-5 : 6 : 7 : 8-tetrahydro-1-naphthylamine** (4-Nitro-ar-tetrahydro- $\alpha$ -naphthylamine).Pale yellow needles from EtOH.Aq. M.p. 116°. Sol. most org. solvents and dil. acids.  
Less sol. H<sub>2</sub>O.Acetyl: needles from EtOH.Aq. or H<sub>2</sub>O. M.p. 178°. Sol. org. solvents. Mod. sol. H<sub>2</sub>O.Green, Rowe, *J. Chem. Soc.*, 1918, 113, 959.**1-Nitro-5 : 6 : 7 : 8-tetrahydro-2-naphthylamine** (1-Nitro-ar-tetrahydro- $\beta$ -naphthylamine) $C_{10}H_{12}O_2N_2$ 

MW, 192

Red needles from EtOH. M.p. 96°.

Acetyl: needles from Et<sub>2</sub>O. M.p. 128–9°.Schroeter *et al.*, *Ann.*, 1922, 426, 67.**3-Nitro-5 : 6 : 7 : 8-tetrahydro-2-naphthylamine** (3-Nitro-ar-tetrahydro- $\beta$ -naphthylamine).Red needles from EtOH. M.p. 125–7°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, toluene.N-Me: C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub>. MW, 206. Fine red needles from EtOH. M.p. 113–15°. Sol. usual org. solvents. Acetyl: powder. M.p. 107–108.5°. Sol. most org. solvents.

Acetyl: yellow needles from EtOH. M.p. 135°.

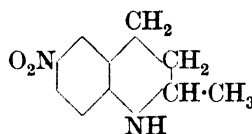
See previous reference.

**4-Nitro-2 : 3 : 5 : 6-tetramethylphenol****4-Nitro-5 : 6 : 7 : 8-tetrahydro-2-naphthylamine** (4-Nitro-ar-tetrahydro- $\beta$ -naphthylamine).

Yellow cryst. powder. M.p. 55°. Sol. most org. solvents.

Acetyl: needles from EtOH. M.p. 194°. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

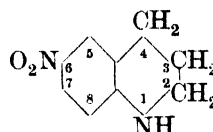
See previous reference.

**6-Nitro-1 : 2 : 3 : 4-tetrahydroquinaldine** $C_{10}H_{12}O_2N_2$ 

MW, 192

dl-.

Brownish-red cryst. M.p. 130–2°.

Stoermer, *Ber.*, 1898, 31, 2540.**6-Nitro-1 : 2 : 3 : 4-tetrahydroquinoline** $C_9H_{10}O_2N_2$ 

MW, 178

Dark yellow needles with bluish reflex. M.p. 163–4°.

N-Nitroso: yellow needles. M.p. 154–5°.

Stoermer, *Ber.*, 1898, 31, 2537.v. Dorp, *Rec. trav. chim.*, 1904, 23, 307.**7-Nitro-1 : 2 : 3 : 4-tetrahydroquinoline.**Yellowish-red cryst. from Et<sub>2</sub>O-pet. ether. M.p. 90°. Very sol. EtOH, Et<sub>2</sub>O.

B.HCl: needles from EtOH. M.p. 203°. Spar. sol. EtOH.

N-Me: 7-nitrokairolin. C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 192. Red needles from EtOH.Aq. M.p. 93–4°. Sol. usual solvents and conc. acids.

N-Benzoyl: m.p. 141°. Spar. sol. EtOH.

N-Nitroso: m.p. 118–20°. Spar. sol. EtOH.

v. Braun, Grabowski, Rawicz, *Ber.*, 1913, 46, 3170, 3173.Feer, Koenigs, *Ber.*, 1885, 18, 2390.**8-Nitro-1 : 2 : 3 : 4-tetrahydroquinoline.**

Red needles. M.p. 82–3°.

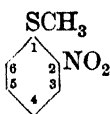
N-Nitroso: reddish-brown needles from EtOH. M.p. 99–100°.

Stoermer, *Ber.*, 1898, 31, 2537.**4-Nitro-2 : 3 : 5 : 6-tetramethylphenol.**

See 4-Nitrodurenol.



## o-Nitrothioanisole

 $C_7H_7O_2NS$ 

MW, 169

Yellow needles from EtOH or  $H_2O$ . M.p.  $64-5^\circ$ . Sol.  $H_2O$ , EtOH, AcOH, AcOEt,  $C_6H_6$ . Less sol. EtOH.Aq.  $D_4^{78.2}$  1.2626.  $n_D^{78.2}$  1.62458.  $B, AgNO_3$ : yellow leaflets. M.p.  $122^\circ$ .

Brand, *Ber.*, 1909, **42**, 3988.Claasz, *Ber.*, 1912, **45**, 1022.Brand, Kranz, *J. prakt. Chem.*, 1927, **115**, 143.

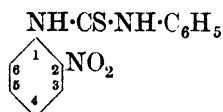
## p-Nitrothioanisole.

Yellow leaflets from ligroin, plates from MeOH. M.p.  $72^\circ$ .  $D_4^{80.1}$  1.2391.  $n_D^{80.1}$  1.64008.

See last reference above and also

Mayer, *Ber.*, 1909, **42**, 3050.Brand, Wirsing, *Ber.*, 1912, **45**, 1763.

## o-Nitrothiocarbanilide

 $C_{13}H_{11}O_2N_3S$ 

MW, 273

Yellow cryst. from AcOH. M.p.  $142^\circ$  ( $188^\circ$ ). Sol. EtOH. Insol. Et<sub>2</sub>O.

Arndt, Rosenau, *Ber.*, 1917, **50**, 1258.Dyson, *J. Chem. Soc.*, 1934, 176.

## m-Nitrothiocarbanilide.

Yellow needles. M.p.  $155^\circ$ . Sol. warm EtOH. Spar. sol.  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ .

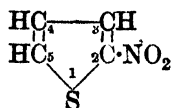
Bruckner, *Ber.*, 1874, **7**, 1235.Losanitsch, *Ber.*, 1881, **14**, 2365.

## p-Nitrothiocarbanilide.

Pale yellow plates or prisms from EtOH. M.p.  $141^\circ$  ( $175^\circ$ ).

Hunter, Jones, *J. Chem. Soc.*, 1930, 2206.Dyson, *J. Chem. Soc.*, 1934, 176.

## 2-Nitrothiophene

 $C_4H_3O_2NS$ 

MW, 129

Cryst. M.p.  $46.5^\circ$ . B.p.  $224-5^\circ$ . Turns red slowly in air. Sol. hot alkalis with deep red-

dish-brown col. Volatile in steam. Et<sub>2</sub>O sol. causes blisters on skin.

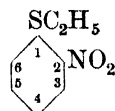
Steinkopf, *Ann.*, 1914, **403**, 18.Steinkopf, Lützkendorf, D.R.P., 255,394, (*Chem. Zentr.*, 1913, I, 476).Babasinian, *Organic Syntheses*, 1934, XIV, 76.

## 3-Nitrothiophene.

Needles from  $H_2O$ , leaflets from EtOH. M.p.  $77^\circ$  ( $75-7^\circ$ ). B.p.  $225^\circ$ ,  $95^\circ/12$  mm.

Steinkopf, Höpner, *Ann.*, 1933, **501**, 183.Rinkes, *Rec. trav. chim.*, 1933, **52**, 538.

## o-Nitrothiophenetole

 $C_8H_9O_2NS$ 

MW, 183

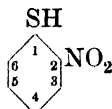
Oil. B.p.  $149-50^\circ/15$  mm.Foster, Reid, *J. Am. Chem. Soc.*, 1924, **46**, 1939.

## p-Nitrothiophenetole.

Yellow needles from 80% AcOH. M.p.  $44^\circ$ .

Brand, Wirsing, *Ber.*, 1913, **46**, 823.Waldron, Reid, *J. Am. Chem. Soc.*, 1923, **45**, 2402.

## o-Nitrothiophenol

 $C_6H_5O_2NS$ 

MW, 155

Yellow needles from  $CCl_4$ . M.p.  $57-8^\circ$ .

S-Me ether: see o-Nitrothioanisole.

S-Et ether: see o-Nitrothiophenetole.

S-Propyl ether:  $C_9H_{11}O_2NS$ . MW, 197. Oil. B.p.  $172-4^\circ/7$  mm.

S-Allyl ether:  $C_9H_9O_2NS$ . MW, 195. Yellow needles from EtOH. M.p.  $54^\circ$ .

S-Phenyl ether: see 2-Nitrodiphenyl sulphide.

S-o-Tolyl ether: see 2'-Nitro-2-methyldiphenyl sulphide.

Brand, *Ber.*, 1909, **42**, 3465.Claasz, *Ber.*, 1912, **45**, 2427.Blanksma, *Rec. trav. chim.*, 1901, **20**, 400.Foster, Reid, *J. Am. Chem. Soc.*, 1924, **46**, 1937.

## p-Nitrothiophenol.

Cryst. from Et<sub>2</sub>O,  $CHCl_3$  or  $Me_2CO$ . M.p.  $77^\circ$ . Very sol. Et<sub>2</sub>O,  $CHCl_3$ ,  $Me_2CO$ . Mod. sol. hot  $H_2O$ , EtOH. Spar. sol. AcOH, ligroin.

S-Me ether: see p-Nitrothioanisole.

S-Et ether: see p-Nitrothiophenetole.

S-Propyl ether: brown oil.  $D_{25}^{25}$  1.1963.

S-Isopropyl ether:  $C_9H_{11}O_2NS$ . MW, 197. Yellow needles from 80% AcOH. M.p. 44.5°.

S-Allyl ether: yellow plates from EtOH. M.p. 38–9°.

S-Butyl ether:  $C_{10}H_{13}O_2NS$ . MW, 211. Brown oil.  $D_{25}^{25}$  1.1625.

S-Isobutyl ether: brown oil.  $D_{25}^{25}$  1.1573.

S-Isoamyl ether:  $C_{11}H_{15}O_2NS$ . MW, 225. Brown oil.  $D_{25}^{25}$  1.1335.

S- $\beta$ -Hydroxyethyl ether: yellow needles from 80% AcOH. M.p. 59°.

S- $\beta$ -Bromoethyl ether: yellow plates from AcOH. M.p. 58°.

S-Phenyl ether: see 4-Nitrodiphenyl sulphide.

S-p-Tolyl ether: see 4'-Nitro-4-methyldiphenyl sulphide.

S-Benzyl ether: yellow plates from 80% AcOH. M.p. 123°.

S-Phenacyl ether: yellow plates from 80% AcOH. M.p. 118°.

S-Cyclohexyl ether: yellow needles from EtOH. M.p. 56–7°.

S-Benzoyl: yellow plates from 50% AcOH. M.p. 123–7°.

Foster, Reid, *J. Am. Chem. Soc.*, 1924, **46**, 1939.

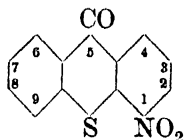
Waldron, Reid, *J. Am. Chem. Soc.*, 1923, **45**, 2402.

Fromm, Wittmann, *Ber.*, 1908, **41**, 2267.

Brand, Wirsing, *Ber.*, 1913, **46**, 822.

M.L.B., D.R.P., 228,868, (*Chem. Zentr.*, 1911, I, 50).

### 1-Nitrothioxanthone



$C_{13}H_7O_3NS$

MW, 257

Yellow needles from  $C_6H_6$ , prisms from xylene. M.p. 216–18° (215°). Spar. sol.  $C_6H_6$ , AcOH. Very spar. sol. EtOH.

Bayer, D.R.P., 228,756, (*Chem. Zentr.*, 1910, II, 1842).

Mayer, *Ber.*, 1909, **42**, 3062.

### 2-Nitrothioxanthone.

Leaflets from AcOH. M.p. 247°.

Mayer, *Ber.*, 1909, **42**, 3067.

### 3-Nitrothioxanthone.

Light brown plates from AcOH. M.p. 219–21°. Spar. sol. AcOH,  $C_6H_6$ . Very spar. sol. EtOH.

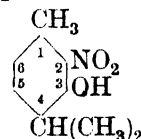
See previous reference.

### 4-Nitrothioxanthone.

Plates from AcOH. M.p. 237°. Mod. sol.  $C_6H_6$ . Spar. sol. EtOH.

See previous reference.

### 2-Nitrothymol



$C_{10}H_{13}O_3N$

MW, 195

Cryst. from EtOH or  $H_2O$ . M.p. 119°. Volatile in steam.

Et ether:  $C_{12}H_{17}O_3N$ . MW, 223. Yellow liq. Volatile in steam.

Robertson, *J. Chem. Soc.*, 1908, **93**, 793 (Footnote).

Gaebel, *Ber.*, 1902, **35**, 2797.

### 6-Nitrothymol.

Needles with bluish fluor. from ligroin- $C_6H_6$ . M.p. 140–2° (137°).

Et ether: yellow plates from EtOH. M.p. 60–1°. Sol. EtOH,  $Et_2O$ . Volatile in steam.

Robertson, Briscoe, *J. Chem. Soc.*, 1912, **101**, 1968.

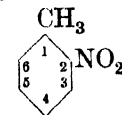
Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2042.

Kehrmann, Schön, *Ann.*, 1909, **310**, 107.

### 6-Nitro-o-tolidine.

See 6-Nitro-4 : 4'-diamino-3 : 3'-dimethyldiphenyl.

### o-Nitrotoluene



$C_7H_7O_2N$

MW, 137

( $\alpha$ -) Transparent needles. M.p. –9.55°. ( $\beta$ -) Opaque snow-like cryst. M.p. –3.85°.  $\beta$ -Form more stable at low temps. and in presence of  $HNO_3$ . B.p. 222.3° (219–219.5°/762 mm.).  $D_{15}^{15}$  1.1622.  $n_D^{20}$  1.54739.  $KMnO_4 \rightarrow$  o-nitrobenzoic acid.  $NaOH \rightarrow$  anthranilic acid. Red.  $\rightarrow$  o-toluidine.  $NaHg \rightarrow$  o-azotoluene and o-hydrazotoluene.

$C_7H_7O_2N, AlCl_3$ : hygroscopic yellow needles. M.p. 99.5°.

$C_7H_7O_2N, AlBr_3$ : yellow needles. M.p. 90°.

$C_7H_7O_2N, SbCl_3$ : needles. M.p.  $34.5^\circ$ .  
 $C_7H_7O_2N, SbBr_3$ : needles. M.p.  $32^\circ$  decomp.

Hennaut-Roland, *Bull. soc. chim. Belg.*, 1933, **42**, 80.

Gibson, Duckham, Fairburn, *J. Chem. Soc.*, 1922, **121**, 270.

Clark, Crozier, *Chem. Abstracts*, 1926, **20**, 389.

Hollemann, Vermeulen, *Rec. trav. chim.*, 1914, **33**, 10.

Kohn, *Monatsh.*, 1910, **31**, 745.

### m-Nitrotoluene.

M.p.  $16^\circ$ . B.p.  $227.2-227.5^\circ/736$  mm.  $D_4^{20}$  1.15712.  $n_D^{21}$  1.5470.  $CrO_3$  or  $KMnO_4 \rightarrow$  m-nitrobenzoic acid. Red.  $\rightarrow$  m-toluidine.  $NaHg \rightarrow$  m-azotoluene and m-hydrazotoluene.  
 $C_7H_7O_2N, AlCl_3$ : hygroscopic needles. M.p.  $99.5^\circ$ .

$C_7H_7O_2N, AlBr_3$ : pale yellow prisms. M.p.  $96^\circ$ .

See first reference above and also

Clarke, Taylor, *Organic Syntheses*, 1923, **III**, 91.

Gibson, Duckham, Fairburn, *J. Chem. Soc.*, 1922, **121**, 270.

### p-Nitrotoluene.

Cryst. from EtOH or  $Et_2O$ . M.p.  $54.5^\circ$  ( $52^\circ$ ). B.p.  $237.7^\circ$ ,  $104.5^\circ/9$  mm.,  $64-5^\circ/0.052$  mm. Spar. sol.  $H_2O$ . Mod. sol. MeOH, EtOH. Sol.  $Et_2O$ ,  $Me_2CO$ , AcOEt,  $CS_2$ ,  $CHCl_3$ ,  $CCl_4$ ,  $C_6H_6$ , toluene, Py.  $D_4^{20}$  1.1038.  $CrO_3$  or  $KMnO_4 \rightarrow$  p-nitrobenzoic acid.  $NaHg$  in EtOH  $\rightarrow$  p-azoxytoluene and p-azotoluene.  $Fe + HCl \rightarrow$  p-toluidine.

$C_7H_7O_2N, AlCl_3$ : hygroscopic plates. M.p.  $109^\circ$ .

$C_7H_7O_2N, AlBr_3$ : cryst. M.p.  $88^\circ$ .

$C_7H_7O_2N, SbCl_3$ : m.p.  $7.5^\circ$  decomp.

Minnis, U.S.P., 1,920,517, (*Chem. Abstracts*, 1933, **27**, 4819).

Lewin, *J. prakt. Chem.*, 1930, **126**, 219.

Desvergnès, *Chem. Abstracts*, 1925, **19**, 3258.

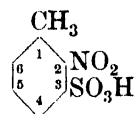
Gibson, Duckworth, Fairburn, *J. Chem. Soc.*, 1922, **121**, 270.

Hollemann, Vermeulen, *Rec. trav. chim.*, 1914, **33**, 10.

### $\omega$ -Nitrotoluene.

See Phenylnitromethane.

### o-Nitrotoluene-3-sulphonic Acid



$C_7H_7O_5NS$  MW, 217

Ba salt: leaflets +  $2H_2O$ . Spar. sol.  $H_2O$ .

Chloride:  $C_7H_6O_4NClS$ . MW, 235.5. M.p.  $58-5^\circ$ .

Amide:  $C_7H_8O_4N_2S$ . MW, 216. M.p.  $163.5^\circ$ .

Foth, *Ann.*, 1885, **230**, 308.

v. Pechmann, *Ann.*, 1874, **173**, 214.

### o-Nitrotoluene-4-sulphonic Acid.

Long pale yellow needles from  $H_2O$ . M.p.  $92^\circ$ . Very hygroscopic.

Phenyl ester:  $C_{13}H_{11}O_5NS$ . MW, 293. Needles from EtOH. M.p.  $59-60^\circ$ . Sol.  $Me_2CO$ ,  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ . Mod. sol. hot EtOH, ligroin.

p-Nitrophenyl ester: needles. M.p.  $113-14^\circ$ . Sol.  $CHCl_3$ . Mod. sol.  $Et_2O$ . Spar. sol. EtOH, ligroin.

o-Tolyl ester: needles. M.p.  $68-9^\circ$ .

m-Tolyl ester: m.p.  $63^\circ$ .

p-Tolyl ester: m.p.  $95^\circ$ .

Fluoride:  $C_7H_6O_4NFS$ . MW, 219.5. Pale yellow cryst. from  $CS_2$ . M.p.  $48-9^\circ$ . Sol.  $Et_2O$ ,  $Me_2CO$ , AcOEt,  $C_6H_6$ , pet. ether. Mod. sol. EtOH. Spar. sol. cold  $CS_2$ . Insol.  $H_2O$ . Spar. volatile in steam.

Chloride:  $C_7H_6O_4NClS$ . MW, 235.5. Plates from  $Et_2O$ . M.p.  $36^\circ$ .

Amide:  $C_7H_8O_4N_2S$ . MW, 216. Leaflets from  $H_2O$ . M.p.  $144.5^\circ$ . Sol. boiling  $H_2O$ , hot EtOH.

Methylamide:  $C_8H_{10}O_4N_2S$ . MW, 230. Pale yellow prisms from EtOH. M.p.  $93^\circ$ .

Ethylamide:  $C_9H_{12}O_4N_2S$ . MW, 244. Pale yellow prisms from EtOH. M.p.  $87^\circ$ .

Anilide:  $C_{13}H_{12}O_4N_2S$ . MW, 292. Cryst. from EtOH.Aq. M.p.  $109^\circ$ . Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Spar. sol. ligroin. Insol.  $H_2O$ .

o-Toluidide:  $C_{14}H_{14}O_4N_2S$ . MW, 306. Cryst. from EtOH. M.p.  $128^\circ$ .

p-Toluidide: m.p.  $130-1^\circ$ .

p-Anisidide: needles from AcOH or EtOH. M.p.  $81^\circ$ . Sol. EtOH,  $Me_2CO$ , AcOH,  $Et_2O$ ,  $C_6H_6$ , hot ligroin. Insol.  $H_2O$ .

m-Phenetidide: needles from AcOH. M.p.  $88^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ ,  $Et_2O$ , ligroin.

p-Phenetidide: cryst. M.p.  $128^\circ$ . Acetyl: plates. M.p.  $172^\circ$ .

Benzylamide: pale yellow plates from EtOH. M.p.  $94^\circ$ .

1-Naphthylamide: cryst. from boiling  $C_6H_6$ . M.p. 153°. Sol.  $CHCl_3$ . Mod. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol. boiling  $H_2O$ .

2-Naphthylamide: cryst. from  $C_6H_6$ . M.p. 161°.

Reverdin, Crépieux, *Bull. soc. chim.*, 1901, 25, 1045; 1902, 27, 745.

Fichter, Bernouilli, *Ber.*, 1909, 42, 4309.

Steinkopf *et al.*, *J. prakt. Chem.*, 1927, 117, 25.

Hirwe, Jambhekar, *J. Indian Chem. Soc.*, 1933, 10, 48.

#### o-Nitrotoluene-5-sulphonic Acid.

Chloride: prisms from  $Et_2O$ . M.p. 50°. Sol.  $Et_2O$ , AcOH. Spar. sol. EtOH, ligroin.

Amide: needles. M.p. 133.5°. Sol. EtOH. Spar. sol. cold  $H_2O$ .

Foth, *Ann.*, 1885, 230, 305.

#### o-Nitrotoluene-6-sulphonic Acid.

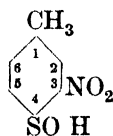
Yellow needles +  $2H_2O$  from  $H_2O$ . M.p. 127°.

Amide: plates from  $H_2O$ . M.p. 165°.

Ba salt: red needles from  $H_2O$ .

Hirwe, Jambhekar, *J. Indian Chem. Soc.*, 1934, 11, 242.

#### m-Nitrotoluene-4-sulphonic Acid



$C_7H_7O_5NS$

MW, 217

Chloride:  $C_7H_6O_4NCIS$ . MW, 235.5. Cryst. from  $C_6H_6$ -pet. ether. M.p. 98-9°. Sol.  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. pet. ether.

Amide:  $C_7H_8O_4N_2S$ . MW, 216. Leaflets from EtOH.Aq. M.p. 170°.

o-Anisidide: yellow needles from AcOH. M.p. 135°.

Zincke, Röse, *Ann.*, 1914, 406, 134.

Heller, *J. prakt. Chem.*, 1929, 121, 193.

#### m-Nitrotoluene-5-sulphonic Acid.

K salt: cryst. from EtOH.

Ba salt: leaflets +  $2H_2O$  from  $H_2O$ .

v. Dorssen, *Rec. trav. chim.*, 1910, 29, 379.

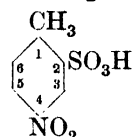
#### m-Nitrotoluene-6-sulphonic Acid.

o-Toluidide: cryst. from AcOH. M.p. 177°.

p-Phenetidide: m.p. 127°.

Schuloff, Pollak, Reisz, *Ber.*, 1929, 62, 1849.

#### p-Nitrotoluene-2-sulphonic Acid



$C_7H_7O_5NS$

MW, 217

Plates +  $2H_2O$  from  $H_2O$ . M.p. 133.5°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

Phenyl ester:  $C_{13}H_{11}O_5NS$ . MW, 293. Cryst. from EtOH. M.p. 64°. Alc. alkalis  $\rightarrow$  orange-yellow sol.  $\rightarrow$  blue on standing.

p-Nitrophenyl ester: m.p. 195°.

Fluoride:  $C_7H_6O_4NFS$ . MW, 219.5. Prisms from pet. ether. M.p. 57-8°. Sol.  $Et_2O$ ,  $Me_2CO$ , AcOEt, EtOH,  $C_6H_6$ .

Chloride:  $C_7H_6O_4NCIS$ . MW, 235.5. Plates or prisms from  $Et_2O$ -pet. ether. M.p. 46-7° (43-44.5°). B.p. 183-5°/10 mm.

Amide:  $C_7H_8O_4N_2S$ . MW, 216. Needles from  $H_2O$ . M.p. 186°. Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ . Benzoyl: prisms from EtOH. M.p. 130°. Sol. boiling EtOH. Spar. sol. hot  $H_2O$ ,  $Et_2O$ .

Anilide:  $C_{13}H_{12}O_4N_2S$ . MW, 292. Rhombic cryst. from  $Et_2O$ . M.p. 148°. Sol. AcOH, hot EtOH. Spar. sol.  $C_6H_6$ . Very spar. sol.  $Et_2O$ .

Steinkopf *et al.*, *J. prakt. Chem.*, 1927, 117, 36.

Walter, *Chem. Ind.*, 1887, 10, 309.

Green, Marsden, Scholefield, *J. Chem. Soc.*, 1904, 85, 1432.

Ullmann, Lehner, *Ber.*, 1905, 38, 736.

Jenssen, *Ann.*, 1874, 172, 232.

Osakeyhtio, Norwegian P., 30,325, (*Chem.*

*Abstracts*, 1920, 14, 2936).

Hintikka, Canadian P., 200,291, (*Chem.*

*Abstracts*, 1920, 14, 2344).

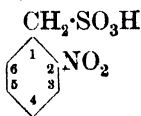
#### p-Nitrotoluene-3-sulphonic Acid.

K salt: pale yellow cryst. powder.

Chloride: pale brown oil.

Coffey, *J. Chem. Soc.*, 1926, 3222.

#### o-Nitrotoluene- $\alpha$ -sulphonic Acid (2-Nitrobenzylsulphonic acid)



$C_7H_7O_5NS$

MW, 217

Cryst. Very hygroscopic.

Na salt: needles +  $H_2O$  from EtOH.

*Ba salt*: cryst. + 3H<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.  
*Amide*: C<sub>7</sub>H<sub>8</sub>O<sub>4</sub>N<sub>2</sub>S. MW, 216. Cryst.  
 M.p. 137°.

Fischer, D.R.P., 48,722.  
 Weiss, Reiter, *Ann.*, 1905, 355, 177.  
 Marckwald, Frahn, *Ber.*, 1898, 31,  
 1855.  
 Clutterbuck, Cohen, *J. Chem. Soc.*, 1923,  
 123, 2512.

**m-Nitrotoluene- $\alpha$ -sulphonic Acid (3-Nitrobenzylsulphonic acid).**

Cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 74°. Mod.  
 sol. H<sub>2</sub>O, EtOH. Spar. sol. other solvents.

*Me ester*: C<sub>8</sub>H<sub>8</sub>O<sub>5</sub>NS. MW, 231. Cryst.  
 M.p. 99–100° (77°). Sol. EtOH, Et<sub>2</sub>O. Insol.  
 H<sub>2</sub>O.

*Chloride*: C<sub>7</sub>H<sub>6</sub>O<sub>4</sub>NCIS. MW, 235.5. Rhom-  
 bic cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 100°.

*Amide*: cryst. from H<sub>2</sub>O. M.p. 159° de-  
 comp. Sol. warm H<sub>2</sub>O.

*Methylamide*: C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>S. MW, 230. M.p.  
 106–7°.

*Dimethylamide*: C<sub>9</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub>S. MW, 244.  
 M.p. 118–19°.

*Ethylamide*: C<sub>9</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub>S. MW, 244. M.p.  
 90–1°.

Purgotti, Monti, *Gazz. chim. ital.*, 1900,  
 30, ii, 247.

Clutterbuck, Cohen, *J. Chem. Soc.*, 1923,  
 123, 2512.

Ingold, Ingold, Shaw, *J. Chem. Soc.*, 1927,  
 824.

**p-Nitrotoluene- $\alpha$ -sulphonic Acid (4-Nitrobenzylsulphonic acid).**

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 71°. Very sol.  
 H<sub>2</sub>O, EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. Et<sub>2</sub>O.

*Me ester*: m.p. 113°.

*Chloride*: m.p. 90°.

*Amide*: prisms from hot H<sub>2</sub>O. M.p. 204°  
 (200°).

*Methylamide*: m.p. 143–5°.

*Dimethylamide*: m.p. 167°.

*Ethylamide*: m.p. 110–11°.

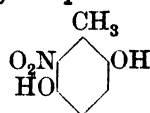
*Anilide*: needles from H<sub>2</sub>O. M.p. 220°  
 decomp.

*Hydrazide*: cryst. from H<sub>2</sub>O. M.p. 87°  
 decomp.

*Phenylhydrazide*: m.p. 156° decomp.

See last two references above and also  
 Weiss, Reiter, *Ann.*, 1905, 355, 177.  
 Mohr, *Ann.*, 1883, 221, 216.

**6-Nitrotoluhydroquinone**

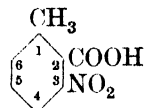


C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>N MW, 169

Red needles from Et<sub>2</sub>O–pet. ether. M.p.  
 117–18°. Aq. alkalis → violet sols.

Cohen, Marshall, *J. Chem. Soc.*, 1904,  
 85, 528.

**3-Nitro-o-toluic Acid**



C<sub>8</sub>H<sub>7</sub>O<sub>4</sub>N MW, 181

Pale yellowish needles. M.p. 151–2°.

*Me ester*: C<sub>9</sub>H<sub>9</sub>O<sub>5</sub>N. MW, 195. Needles  
 and leaflets. M.p. 50°.

*Chloride*: C<sub>8</sub>H<sub>6</sub>O<sub>3</sub>NCI. MW, 199.5. Cryst.  
 from ligroin. M.p. 41°.

*Amide*: C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub>. MW, 180. Needles.  
 M.p. 158°.

*Nitrile*: C<sub>8</sub>H<sub>6</sub>O<sub>2</sub>N<sub>2</sub>. MW, 162. Plates from  
 C<sub>6</sub>H<sub>6</sub>. M.p. 109–10°. Sol. usual solvents.  
 Sublimes slowly in leaflets.

*Anhydride*: plates from EtOH. M.p. 174°.

Gabriel, Thieme, *Ber.*, 1919, 52, 1074,  
 1089.

**4-Nitro-o-toluic Acid.**

Needles from EtOH.Aq. M.p. 179°. Very  
 sol. EtOH. Spar. sol. boiling H<sub>2</sub>O. Spar.  
 volatile in steam.

*Me ester*: prisms from MeOH. M.p. 69°.

*Chloride*: needles from CHCl<sub>3</sub>. M.p. 59–60°.

*Amide*: needles from boiling H<sub>2</sub>O. M.p.  
 173–4°. Sol. Me<sub>2</sub>CO, hot H<sub>2</sub>O, EtOH, AcOH.  
 Spar. sol. Et<sub>2</sub>O, ligroin.

*Methylamide*: C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 194.  
 Needles from EtOH.Aq. M.p. 160°. Sol.  
 EtOH. Mod. sol. H<sub>2</sub>O.

*Dimethylamide*: C<sub>10</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 208.  
 Cryst. from EtOH. M.p. 105–6°. Mod. sol.  
 H<sub>2</sub>O.

*Nitrile*: needles from 95% EtOH. M.p.  
 105°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, hot EtOH.  
 Less sol. hot H<sub>2</sub>O, Et<sub>2</sub>O, AcOH, CS<sub>2</sub>. Spar.  
 sol. pet. ether.

Ruggli, Meyer, *Helv. Chim. Acta*, 1922, 5,  
 58.

Jacobsen, Wierss, *Ber.*, 1883, 16, 1958.  
 v. Scherpenzeel, *Rec. trav. chim.*, 1901,  
 20, 174.

Landsberger, *Ber.*, 1898, 31, 2880.

**5-Nitro-*o*-toluic Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $152^\circ$ . Very sol. EtOH, hot  $\text{H}_2\text{O}$ .

*Nitrile*: yellow needles from EtOH. M.p.  $113\text{--}15^\circ$ ; leaflets by sublimation, m.p.  $110^\circ$ . Sol.  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, hot  $\text{H}_2\text{O}$ .

Gabriel, Thieme, *Ber.*, 1919, **52**, 1089.

Mayer, *J. prakt. Chem.*, 1915, **92**, 142.

**6-Nitro-*o*-toluic Acid.**

Cryst. M.p.  $184\text{--}184.5^\circ$ .

*Me ester*: needles from MeOH. M.p.  $66^\circ$ .

*Chloride*: cryst. from  $\text{CHCl}_3$ . M.p.  $68\text{--}68.5^\circ$ .

*Amide*: needles from  $\text{H}_2\text{O}$ . M.p.  $163^\circ$ .

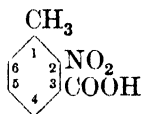
*Methylamide*: needles from  $\text{H}_2\text{O}$ . M.p.  $131\text{--}2^\circ$ .

*Dimethylamide*: yellowish cryst. from EtOH. M.p.  $69.5\text{--}70^\circ$ .

*Nitrile*: cryst. M.p.  $69.5^\circ$ .

v. Scherpenzeel, *Rec. trav. chim.*, 1901, **20**, 172.

Noelting, *Ber.*, 1904, **37**, 1025.

**2-Nitro-*m*-toluic Acid**

$\text{C}_8\text{H}_7\text{O}_4\text{N}$

MW, 181

Prisms from EtOH. M.p.  $223^\circ$  ( $219\text{--}20^\circ$ ). Sol. hot EtOH. Spar. sol.  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_9\text{H}_9\text{O}_4\text{N}$ . MW, 195. Cryst. from MeOH. M.p.  $74^\circ$ .

*Chloride*:  $\text{C}_8\text{H}_7\text{O}_3\text{NCl}$ . MW, 199.5. Cryst. from  $\text{CS}_2$ . Sol. EtOH,  $\text{Et}_2\text{O}$ .

*Amide*:  $\text{C}_8\text{H}_8\text{O}_3\text{N}_2$ . MW, 180. Needles from  $\text{H}_2\text{O}$ , prisms from EtOH. M.p.  $192^\circ$ .

*Methylamide*:  $\text{C}_9\text{H}_{10}\text{O}_3\text{N}_2$ . MW, 194. Plates from  $\text{H}_2\text{O}$ . M.p.  $135\text{--}6^\circ$ . Spar. sol.  $\text{Et}_2\text{O}$ .

*Dimethylamide*:  $\text{C}_{10}\text{H}_{12}\text{O}_3\text{N}_2$ . MW, 208. Cryst. from EtOH. M.p.  $88.5^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Turns red on exposure to light.

*Nitrile*:  $\text{C}_8\text{H}_6\text{O}_2\text{N}_3$ . MW, 162. Needles from EtOH. M.p.  $84^\circ$ .

Jürgens, *Ber.*, 1907, **40**, 4409.

v. Scherpenzeel, *Rec. trav. chim.*, 1901, **20**, 164.

Gabriel, Thieme, *Ber.*, 1919, **52**, 1091.

**4-Nitro-*m*-toluic Acid.**

Cryst. from hot  $\text{H}_2\text{O}$ . M.p.  $134^\circ$ . Hot alkalis  $\rightarrow$  red sols.

*Me ester*: needles from MeOH. M.p.  $78\text{--}9^\circ$ .

*Amide*: needles from EtOH. M.p.  $176\text{--}7^\circ$ . Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ .

*Nitrile*: needles from EtOH. M.p.  $93\text{--}4^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH, AcOH. Less sol.  $\text{C}_6\text{H}_6$ .

Findeklee, *Ber.*, 1905, **38**, 3544.

Reissert, Scherk, *Ber.*, 1898, **31**, 390.

Müller, *Ber.*, 1909, **42**, 430.

Pfeiffer, *Ber.*, 1918, **51**, 559.

**5-Nitro-*m*-toluic Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $174^\circ$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$ . Non-volatile in steam.

*Me ester*: plates from MeOH. M.p.  $84\text{--}5^\circ$ .

*Chloride*: cryst. M.p. about  $100^\circ$ .

*Amide*: needles from  $\text{H}_2\text{O}$ . M.p.  $164\text{--}5^\circ$ . Sol. hot EtOH. Spar. sol.  $\text{Et}_2\text{O}$ .

*Nitrile*: needles from ligroin. M.p.  $104\text{--}5^\circ$ .

Gabriel, Thieme, *Ber.*, 1919, **52**, 1090.

Müller, *Ber.*, 1909, **42**, 433.

**6-Nitro-*m*-toluic Acid.**

Needles from EtOH. M.p.  $219^\circ$  ( $212^\circ$ ). Spar. sol. hot  $\text{H}_2\text{O}$ .  $k = 3.1 \times 10^{-4}$  at  $25^\circ$ . Sublimes.

*Me ester*: needles from EtOH. M.p.  $81\text{--}2^\circ$  ( $72^\circ$ ). Sol. hot EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. hot  $\text{H}_2\text{O}$ .

*Et ester*:  $\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$ . MW, 209. Prisms from EtOH. M.p.  $55^\circ$ . B.p.  $150\text{--}6^\circ/9$  mm. Very sol. hot EtOH.

*Amide*: leaflets or prisms from  $\text{H}_2\text{O}$ . M.p.  $151^\circ$ . Mod. sol. hot  $\text{H}_2\text{O}$ .

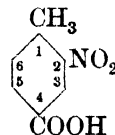
*Nitrile*: prisms from EtOH. M.p.  $80^\circ$ . Insol. cold  $\text{H}_2\text{O}$ .

Suida, *Monatsh.*, 1912, **33**, 1282.

Gabriel, Thieme, *Ber.*, 1919, **52**, 1090.

Beilstein, Kreusler, *Ann.*, 1867, **144**, 168.

Müller, *Ber.*, 1909, **42**, 430.

**2-Nitro-*p*-toluic Acid**

$\text{C}_8\text{H}_7\text{O}_4\text{N}$

MW, 181

Prisms from EtOH. M.p.  $190^\circ$ . Sol. EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_9\text{H}_9\text{O}_4\text{N}$ . MW, 195. Yellowish needles from MeOH. M.p.  $51^\circ$ .

*Et ester*:  $\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$ . MW, 209. Pale yellow cryst.

*Amide*:  $\text{C}_9\text{H}_8\text{O}_3\text{N}_2$ . MW, 180. Needles from  $\text{H}_2\text{O}$ . M.p.  $166\text{--}166.5^\circ$ . Spar. sol.  $\text{C}_6\text{H}_6$ .

**Methylamide**:  $C_9H_{10}O_3N_2$ . MW, 194. M.p. 149°. Sol. EtOH,  $C_6H_6$ , hot  $H_2O$ .

**Dimethylamide**:  $C_{10}H_{12}O_3N_2$ . MW, 208. Cryst. M.p. 49°.

**Nitrile**:  $C_8H_6O_2N_2$ . MW, 162. Pale yellow needles from  $H_2O$ . M.p. 107°. Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>,  $C_6H_6$ . Less sol.  $H_2O$ , EtOH.

Pfeiffer, *Ber.*, 1918, **51**, 563.

Hope, Robinson, *J. Chem. Soc.*, 1911, **99**, 2125.

Pfeiffer, Matton, *Ber.*, 1911, **44**, 1124.

v. Scherpenzeel, *Rec. trav. chim.*, 1901, **20**, 158.

### 3-Nitro-*p*-toluic Acid.

Needles from  $H_2O$ . M.p. 164–5°. Very sol. EtOH. Spar. sol. boiling  $H_2O$ , Et<sub>2</sub>O, CHCl<sub>3</sub>,  $C_6H_6$ .

**Chloride**:  $C_8H_6O_3NCl$ . MW, 199.5. Needles from Et<sub>2</sub>O. M.p. 157°.

**Amide**: needles from  $C_6H_6$  or ligroin. M.p. 153°. Sol. warm  $H_2O$ , EtOH, Et<sub>2</sub>O,  $C_6H_6$ . Insol. ligroin.

**Nitrile**: needles from  $H_2O$ . M.p. 101°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>,  $C_6H_6$ . Insol. ligroin.

Claus, Joachim, *Ann.*, 1891, **266**, 210.

v. Niementowski, Rozański, *Ber.*, 1888, **21**, 1993.

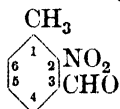
Noyes, *Am. Chem. J.*, 1888, **10**, 474.

Weise, *Ber.*, 1889, **22**, 2429.

### Nitrotoluic Acid sulphonic Acid.

See Nitrosulphotoluic Acid.

### 2-Nitro-*m*-toluic Aldehyde



$C_8H_7O_3N$  MW, 165

Needles from  $H_2O$ . M.p. 64°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH, CHCl<sub>3</sub>,  $C_6H_6$ .

**Oxime**: m.p. 134–5°.

Gilliard, Monnet, Cartier, D.R.P., 113,604, (*Chem. Zentr.*, 1900, II, 751).

Mayer, *Ber.*, 1914, **47**, 406.

### 4-Nitro-*m*-toluic Aldehyde.

Yellow needles. M.p. 44°.

**Oxime**: m.p. 104–5°.

**Phenylhydrazone**: m.p. 141–2°.

See previous references.

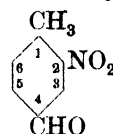
### 6-Nitro-*m*-toluic Aldehyde.

Cryst. from  $H_2O$ . M.p. 64°. Very sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>,  $C_6H_6$ .

**Phenylhydrazone**: m.p. 108°.

Suida, *Monatsh.*, 1912, **33**, 1281.

### 2-Nitro-*p*-toluic Aldehyde



$C_8H_7O_3N$

MW, 165

Pale yellow needles from ligroin. M.p. 48–9° (42–3°). B.p. 140–5°/15 mm. Mod. sol. EtOH, Et<sub>2</sub>O,  $C_6H_6$ .

**α-Oxime**: pale yellow needles from EtOH. M.p. 118–20°. **Acetyl**: cryst. from Me<sub>2</sub>CO.Aq. M.p. 104°.

**β-Oxime**: cryst. from Me<sub>2</sub>CO.Aq. M.p. 135°. **B,HCl**: m.p. 140° decomp.

**Phenylhydrazone**: m.p. 105°.

**p-Nitrophenylhydrazone**: m.p. 233°.

**Diacetyl**: plates from EtOH-ligroin. M.p. 98–98.5°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. ligroin.

Hanzlik, Bianchi, *Ber.*, 1899, **32**, 2286.

Gattermann, *Ann.*, 1906, **347**, 354.

Brady, Cosson, Roper, *J. Chem. Soc.*, 1925, 2431.

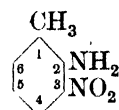
Wahl, *Compt. rend.*, 1934, **198**, 2107.

### 3-Nitro-*p*-toluic Aldehyde.

**Oxime**: needles from  $C_6H_6$ -ligroin. M.p. 128°.

M.L.B., D.R.P., 107,095, (*Chem. Zentr.*, 1900, I, 886).

### 3-Nitro-*o*-toluidine



$C_7H_8O_2N_2$

MW, 152

Orange-yellow prisms from EtOH.Aq. M.p. 97°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>,  $C_6H_6$ . Spar. sol.  $H_2O$ . D<sub>4</sub><sup>20</sup> 1.1900.

**N-Acetyl**: 3-nitroacet-*o*-toluidide. M.p. 158°.

**N-Me**: see 3-Nitro-*N*-methyl-*o*-toluidine.

Harrison, *J. Soc. Chem. Ind.*, 1935, **54**, 283T.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1171.

### 4-Nitro-*o*-toluidine.

Yellow prisms from EtOH. M.p. 107°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Spar. sol.  $H_2O$ .

**N-Formyl**: m.p. 178–9°.

**N-Acetyl**: 4-nitroacet-*o*-toluidide. M.p. 151°. Trimorphous.

**N-m-Nitrobenzoyl**: m.p. 193°.

**N-p-Nitrobenzoyl**: m.p. 214°.

**N-Benzenesulphonyl**: m.p. 172°.

N-1-Naphthalenesulphonyl: m.p. 244°.  
 N-2-Naphthalenesulphonyl: m.p. 229–30°.  
 N-Me: see 4-Nitro-*N*-methyl-*o*-toluidine.  
 N-Di-Me: see 4-Nitro-*N*-dimethyl-*o*-toluidine.

N-Et: see 4-Nitro-*N*-ethyl-*o*-toluidine.  
 N-Di-Et: see 4-Nitro-*N*-diethyl-*o*-toluidine.  
 N-Furfurylidene: m.p. 153° (75°).  
 N-Benzylidene: m.p. 116°.  
 N-*o*-Nitrobenzylidene: m.p. 155°.  
 N-*m*-Nitrobenzylidene: m.p. 185°.  
 N-*p*-Nitrobenzylidene: m.p. 227°.

Pomeranz, D.R.P. 289,454, (*Chem. Zentr.*, 1916, I, 275).

Ullmann, Grether, *Ber.*, 1902, **35**, 337.

Schiff, Vanni, *Ann.*, 1892, **268**, 322.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1333.

McGookin, *J. Chem. Soc.*, 1934, 1743.

### 5-Nitro-*o*-toluidine.

Yellow needles from H<sub>2</sub>O or EtOH. M.p. 129°. Sol. EtOH. Spar. sol. H<sub>2</sub>O. D<sub>4</sub><sup>40</sup> 1.1586.

B<sub>2</sub>HCl: m.p. 199–200°.

B<sub>2</sub>HBr: m.p. 240°.

N-Acetyl: 5-nitroacet-*o*-toluidide. Needles from H<sub>2</sub>O. M.p. 201.6° (198°).

N-Chloroacetyl: m.p. 122°.

N-Benzenesulphonyl: m.p. 157–9°.

N-*p*-Toluenesulphonyl: m.p. 174°.

N-Me: see 5-Nitro-*N*-methyl-*o*-toluidine.

N-Di-Me: see 5-Nitro-*N*-dimethyl-*o*-toluidine.

N-Et: see 5-Nitro-*N*-ethyl-*o*-toluidine.

N-Furfurylidene: m.p. 130°.

N-Phenyl: see 4-Nitro-2-methyldiphenylamine.

Harrison, *J. Soc. Chem. Ind.*, 1935, **54**, 283T.

Jansen, *Chem. Zentr.*, 1913, II, 761.

Meisenheimer, Hesse, *Ber.*, 1919, **52**, 1171.

Kenner, Parkin, *J. Chem. Soc.*, 1920, **117**, 859.

Bogert, Cook, *J. Am. Chem. Soc.*, 1906, **28**, 1451.

### 6-Nitro-*o*-toluidine.

Yellow needles from H<sub>2</sub>O, yellow leaflets from EtOH. M.p. 92°. B.p. 305° decomp. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

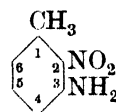
N-Acetyl: 6-nitroacet-*o*-toluidide. Prisms from H<sub>2</sub>O. M.p. 157.5–158°.

N-Benzoyl: m.p. 168°.

N-Di-Me: see 6-Nitro-*N*-dimethyl-*o*-toluidine.

Wheeler, *Am. Chem. J.*, 1910, **44**, 136.

### 2-Nitro-*m*-toluidine



C<sub>7</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub>

MW, 152

Red prisms. M.p. 108°.

N-Acetyl: 2-nitroacet-*m*-toluidide. Prisms from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 126°.

Morton, McGookin, *J. Chem. Soc.*, 1934, 910.

Burton, Kenner, *J. Chem. Soc.*, 1921, **119**, 1052.

### 4-Nitro-*m*-toluidine.

Yellow plates from EtOH.Aq. M.p. 110°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

N-Acetyl: 4-nitroacet-*m*-toluidide. Yellow needles from H<sub>2</sub>O or pet. ether. M.p. 88–9° (86–7°).

N-Benzoyl: m.p. about 83°.

N-Benzenesulphonyl: m.p. 137–8°.

N-Me: see 4-Nitro-*N*-methyl-*m*-toluidine.

N-Et: see 4-Nitro-*N*-ethyl-*m*-toluidine.

N-Phenyl: see 6-Nitro-3-methyldiphenylamine.

Elson, Gibson, Johnson, *J. Chem. Soc.*, 1929, 2739.

Harrison, *J. Soc. Chem. Ind.*, 1935, **54**, 283T.

Kenner, Parkin, *J. Chem. Soc.*, 1920, **117**, 858.

### 5-Nitro-*m*-toluidine.

Brown needles. M.p. 98°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

N-Acetyl: 5-nitroacet-*m*-toluidide. M.p. 187°.

N-Benzoyl: m.p. 177°.

N-Di-Me: see 5-Nitro-*N*-dimethyl-*m*-toluidine.

Morton, McGookin, *J. Chem. Soc.*, 1934, 910.

### 6-Nitro-*m*-toluidine.

Yellow needles from H<sub>2</sub>O. M.p. 135°. Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O. Non-volatile in steam.

N-Acetyl: 6-nitroacet-*m*-toluidide. Prisms from EtOH. M.p. 103–4°.

N-Me: see 6-Nitro-*N*-methyl-*m*-toluidine.

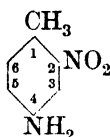
N-Di-Me: see 6-Nitro-*N*-dimethyl-*m*-toluidine.

Wibaut, *Rec. trav. chim.*, 1913, **32**, 287.

Harrison, *J. Soc. Chem. Ind.*, 1935, **54**, 283T.

Kenner, Parkin, *J. Chem. Soc.*, 1920, **117**, 858.



2-Nitro-*p*-toluidine

$C_7H_8O_2N_2$  MW, 152

Yellow needles from  $H_2O$ . M.p.  $78^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ , hot  $EtOH$ . Mod. sol. hot  $H_2O$ . Spar. sol.  $CS_2$ .

*B.HCl*: m.p.  $230-40^\circ$  ( $220^\circ$ ).

*B.HBr, 3H\_2O*: m.p.  $238^\circ$ .

*N-Formyl*: m.p.  $133-4^\circ$ .

*N-Acetyl*: 2-nitroacet-*p*-toluidide. Yellow needles or leaflets from  $H_2O$ . M.p.  $144.5^\circ$ .

*N-Chloroacetyl*: m.p.  $129^\circ$ .

*Succinate*:  $(C_7H_7O_2N_2 \cdot OC \cdot CH_2)_2$ . M.p.  $140^\circ$ .

*N-Benzoyl*: m.p.  $168^\circ$  ( $172^\circ$ ).

*N-Benzenesulphonyl*: m.p.  $160^\circ$ .

*N-p-Toluenesulphonyl*: m.p.  $162-3^\circ$ .

*N-Me*: see 2-Nitro-*N*-methyl-*p*-toluidine.

*N-Di-Me*: see 2-Nitro-*N*-dimethyl-*p*-toluidine.

*N-Et*: see 2-Nitro-*N*-ethyl-*p*-toluidine.

*N-Butyl*:  $C_{11}H_{16}O_2N_2$ . MW, 208. M.p.  $19^\circ$ .

*N-Acetyl*: m.p.  $48-9^\circ$ .

*N-Benzylidene*: m.p.  $77-8^\circ$ .

Noelting, Collin, *Ber.*, 1884, 17, 263.

McGookin, *J. Chem. Soc.*, 1934, 1744.

Morton, McGookin, *ibid.*, 909.

3-Nitro-*p*-toluidine.

Red leaflets from  $EtOH.Aq$ . M.p.  $117^\circ$ . Sol.  $EtOH$ . Spar. sol.  $H_2O$ . Volatile in steam.  $D_4^{25} 1.164$ .  $k = 5.4 \times 10^{-4}$  at  $25^\circ$ .

*B.HCl*: m.p.  $170-1^\circ$ .

*B.HBr*: m.p.  $229-30^\circ$ .

*N-Acetyl*: 3-nitroacet-*p*-toluidide. Yellow needles from pet. ether. M.p.  $96^\circ$ .

*N-Diacetyl*: m.p.  $78^\circ$ .

*N-Chloroacetyl*: m.p.  $122^\circ$  ( $119^\circ$ ).

*N-Trichloroacetyl*: m.p.  $54-5^\circ$ .

*N-Butyryl*: m.p.  $62^\circ$ .

*N-Isovaleryl*: m.p.  $88-9^\circ$ .

*Succinate*:  $(C_7H_7O_2N_2 \cdot OC \cdot CH_2)_2$ . M.p.  $217^\circ$ .

*N-Lactyl*: m.p.  $86-7^\circ$ .

*N-Benzoyl*: m.p.  $146-8^\circ$ .

*N-o-Chlorobenzoyl*: m.p.  $139^\circ$ .

*N-o-Nitrobenzoyl*: m.p.  $198^\circ$ .

*N-m-Nitrobenzoyl*: m.p.  $188.5^\circ$ .

*N-p-Nitrobenzoyl*: m.p.  $171^\circ$ .

*N-p-Toluyyl*: m.p.  $165-6^\circ$ .

*N-Cinnamoyl*: m.p.  $147^\circ$ .

*N-Benzenesulphonyl*: m.p.  $101-2^\circ$ .

*N-p-Toluenesulphonyl*: m.p.  $145-6^\circ$ .

*N-Di-p-toluenesulphonyl*: m.p.  $228^\circ$ .

*N-d-Camphor-β-sulphonyl*: m.p.  $126-8^\circ$ .

*N-o-Chlorobenzylidene*: m.p.  $149^\circ$ .

*N-p-Nitrobenzylidene*: m.p.  $161.5^\circ$ .

*N-2:4-Dinitrobenzylidene*: m.p.  $195^\circ$ .

*N-Me*: see 3-Nitro-*N*-methyl-*p*-toluidine.

*N-Di-Me*: see 3-Nitro-*N*-dimethyl-*p*-toluidine.

*N-Et*: see 3-Nitro-*N*-ethyl-*p*-toluidine.

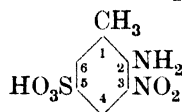
*N-Di-Et*: see 3-Nitro-*N*-diethyl-*p*-toluidine.

Gattermann, *Ber.*, 1885, 18, 1483.

A.G.F.A., D.R.P. 164,130, (*Chem. Zentr.*, 1905, II, 1476).

Ullmann, Gross, *Ber.*, 1910, 43, 2698.

Gindraux, *Helv. Chim. Acta*, 1929, 12, 933.

3-Nitro-*o*-toluidine-5-sulphonic Acid

$C_7H_8O_5N_2S$

MW, 232

Yellow needles from  $H_2O$ .

*K salt*: orange-yellow needles. Sol.  $H_2O$ .

Gnehm, Blumer, *Ann.*, 1899, 304, 105.

Nietzki, Pollini, *Ber.*, 1890, 23, 138.

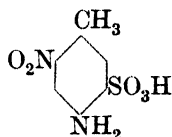
6-Nitro-*o*-toluidine-4-sulphonic Acid.

Needles. Sol. 102.7 parts  $H_2O$  at  $19^\circ$ . Insol. most other solvents.

*K salt*: leaflets. Spar. sol. cold  $H_2O$ .

*Ba salt*: needles +  $2\frac{1}{2}H_2O$ . Spar. sol. cold  $H_2O$ .

Marekwald, *Ann.*, 1893, 274, 350.

6-Nitro-*p*-toluidine-3-sulphonic Acid

$C_7H_8O_5N_2S$

MW, 232

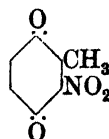
Pale yellow needles from  $H_2O$ . Decomp. on heating. Spar. sol.  $EtOH$ .

*K salt*: orange-red prisms +  $H_2O$ . Spar. sol. cold  $H_2O$ .

*Ba salt*: red cryst.

Foth, *Ann.*, 1885, 230, 300.

## 3-Nitrotoluquinone



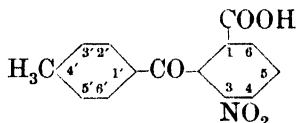
$C_7H_6O_4N$

MW, 167

Ruby red prisms from Et<sub>2</sub>O-pet. ether. M.p. 64–5°.

Cohen, Marshall, *J. Chem. Soc.*, 1904, **85**, 527.

**3-Nitro-2-*p*-toluylbenzoic Acid** (6-Nitro-4'-methylbenzophenone-2-carboxylic acid)



C<sub>15</sub>H<sub>11</sub>O<sub>5</sub>N

MW, 285

Two compounds of this constitution have been described.

(i) Yellow plates from dil. EtOH. M.p. 218–19°.

Mitter, Sarkar, *J. Indian. Chem. Soc.*, 1930, **7**, 625.

(ii) M.p. 123–6° decomp. KOH at 215° → *p*-toluic and *m*-nitrobenzoic acids.

Lawrance, *J. Am. Chem. Soc.*, 1921, **43**, 2579.

**4-Nitro-2-*p*-toluylbenzoic Acid** (5-Nitro-4'-methylbenzophenone-2-carboxylic acid).

Three compounds of this constitution have been described.

(i) Prisms from AcOH. M.p. 171°.

Mitter, Sarkar, *J. Indian Chem. Soc.*, 1930, **7**, 626.

(ii) M.p. 101–5° decomp. KOH at 215° → *p*-toluic and *p*-nitrobenzoic acids.

Lawrance, *J. Am. Chem. Soc.*, 1921, **43**, 2579.

(iii) M.p. 216–217·5° (not pure).

Hayashi, Nakayama, *Chem. Abstracts*, 1934, **28**, 5818.

**5-Nitro-2-*p*-toluylbenzoic Acid** (4-Nitro-4'-methylbenzophenone-2-carboxylic acid).

M.p. 187–91° (not pure).

Hayashi, Nakayama, *Chem. Abstracts*, 1934, **28**, 5819.

**6-Nitro-2-*p*-toluylbenzoic Acid** (3-Nitro-4'-methylbenzophenone-2-carboxylic acid).

M.p. 262–5° decomp. KOH at 215° → *p*-toluic and *o*-nitrobenzoic acids.

Lawrance, *J. Am. Chem. Soc.*, 1921, **43**, 2578.

**3'-Nitro-*p*-toluylbenzoic Acid** (3'-Nitro-4'-methylbenzophenone-2-carboxylic acid).

Cryst. + 1H<sub>2</sub>O. M.p. 205°. Sol. Me<sub>2</sub>CO, AcOEt, warm EtOH. Very spar. sol. Et<sub>2</sub>O, CS<sub>2</sub>, CCl<sub>4</sub>. KOH fusion → benzoic acid.

Dict. of Org. Comp.—III.

*Et ester*: C<sub>17</sub>H<sub>15</sub>O<sub>5</sub>N. MW, 313. Prisms from EtOH. M.p. 122°.

*Chloride*: C<sub>15</sub>H<sub>10</sub>O<sub>4</sub>NCl. MW, 303·5. Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 142°. Stable in air.

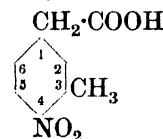
*Amide*: C<sub>15</sub>H<sub>12</sub>O<sub>4</sub>N<sub>2</sub>. MW, 284. Needles from EtOH or C<sub>6</sub>H<sub>6</sub>. Decomp. at 200°.

*Anhydride*: C<sub>30</sub>H<sub>20</sub>O<sub>9</sub>N<sub>2</sub>. MW, 552. Needles from EtOH or toluene. M.p. 203°.

*Mixed anhydride with acetic acid*: C<sub>17</sub>H<sub>13</sub>O<sub>6</sub>N. MW, 327. Cryst. M.p. 145–6°.

Limpricht, *Ann.*, 1898, **299**, 309.

**4-Nitro-*m*-tolylacetic Acid** (4-Nitro-3-methylphenylacetic acid)



C<sub>9</sub>H<sub>9</sub>O<sub>4</sub>N

MW, 195

*Nitrile*: C<sub>9</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub>. MW, 176. Cryst. from H<sub>2</sub>O or EtOH. M.p. 63° (52°). B.p. 200–5°/22 mm. Sol. boiling EtOH. Mod. sol. boiling H<sub>2</sub>O. Alk. KMnO<sub>4</sub> → 4-nitro-*m*-toluic acid.

Barger, Ewins, *J. Chem. Soc.*, 1910, **97**, 2256.

Lifschutz, Jenner, *Ber.*, 1915, **48**, 1740.

**6-Nitro-*m*-tolylacetic Acid** (6-Nitro-3-methylphenylacetic acid).

Cryst. from H<sub>2</sub>O. M.p. 149°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>.

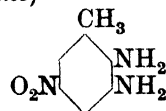
Reissert, Scherk, *Ber.*, 1898, **31**, 391.

**3-Nitro-*p*-tolylacetic Acid** (3-Nitro-4-methylphenylacetic acid).

Needles from H<sub>2</sub>O. M.p. 102°. Sol. EtOH, Et<sub>2</sub>O, CS<sub>2</sub>. Mod. sol. hot H<sub>2</sub>O. Sublimes. KMnO<sub>4</sub> → 3-nitro-*p*-toluic acid.

Claus, Wehr, *J. prakt. Chem.*, 1891, **44**, 90.

**5-Nitro-2 : 3-tolylenediamine** (5-Nitro-2 : 3-diaminotoluene)



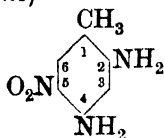
C<sub>7</sub>H<sub>9</sub>O<sub>2</sub>N<sub>3</sub>

MW, 167

Orange-red needles from EtOH.Aq. M.p. 185°. Very sol. hot EtOH, AcOH. Mod. sol. hot H<sub>2</sub>O.

*N : N'-Diacetyl*: yellowish needles from EtOH.Aq. M.p. 234°. Sol. AcOH, hot EtOH. Spar. sol. hot H<sub>2</sub>O.

Kym, Ringer, *Ber.*, 1915, **48**, 1674.

**5-Nitro-2 : 4-tolylenediamine** (5-Nitro-2 : 4-diaminotoluene) $C_7H_9O_2N_3$ 

MW, 167

Yellow needles with violet reflex from  $H_2O$ . M.p.  $154^\circ$ . Sol. hot EtOH. Mod. sol. hot  $H_2O$ . Weak base. Salts decomp. by  $H_2O$ .

4-N-*Me*:  $C_8H_{11}O_2N_3$ . MW, 181. Bronze leaflets from EtOH. M.p.  $168^\circ$ . Sol.  $CHCl_3$ , AcOH, hot  $C_6H_6$ . Spar. sol. ligroin. 2-N-*Acetyl*: yellowish-brown needles from  $H_2O$ . M.p.  $205.5-207^\circ$ . Very sol. hot AcOH. Spar. sol. hot  $H_2O$ .

4-N-*Di-Me*:  $C_9H_{13}O_2N_3$ . MW, 195. Yellow needles from EtOH. M.p.  $155^\circ$ .

N : N'-*Diacetyl*: needles from  $Me_2CO$ . M.p.  $250-1^\circ$ . Sol. about 300 parts  $Me_2CO$ .

N : N'-*Dibenzoyl*: yellow needles from AcOH. M.p.  $245^\circ$ .

N : N'-*Di-benzenesulphonyl*: yellow prisms. M.p.  $185^\circ$ .

N : N'-*Di-p-toluenesulphonyl*: yellow cryst. from AcOH. M.p.  $210^\circ$ .

Brady, Day, Reynolds, *J. Chem. Soc.*, 1929, 2265.

Pinnow, *J. prakt. Chem.*, 1900, **62**, 508.

A.G.F.A., D.R.P., 166,600, (*Chem. Zentr.*, 1906, I, 517).

Ruhemann, *Ber.*, 1881, **14**, 2656.

Morgan, Clayton, *J. Chem. Soc.*, 1910, **97**, 2650.

Ladenburg, *Ber.*, 1875, **8**, 1211.

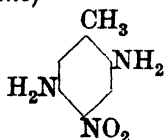
See also Staedel, *Ann.*, 1883, **217**, 155.

**6-Nitro-2 : 4-tolylenediamine** (6-Nitro-2 : 4-diaminotoluene).

Yellowish-orange cryst. from  $C_6H_6$ -pet. ether. M.p.  $130-1^\circ$ .

Brady, Day, Reynolds, *J. Chem. Soc.*, 1929, 2266.

Tiemann, *Ber.*, 1870, **3**, 218.

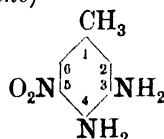
**4-Nitro-2 : 5-tolylenediamine** (4-Nitro-2 : 5-diaminotoluene) $C_7H_9O_2N_3$ 

MW, 167

Red needles with bronze reflex from  $H_2O$ . M.p.  $173^\circ$ .

N : N'-*Diacetyl*: yellowish prisms from AcOH. M.p.  $258^\circ$  decomp. Very spar. sol. usual solvents.

Morgan, Micklethwait, *J. Chem. Soc.*, 1913, **103**, 1398.

**5-Nitro-3 : 4-tolylenediamine** (5-Nitro-3 : 4-diaminotoluene) $C_7H_9O_2N_3$ 

MW, 167

Dark red needles from  $H_2O$ . M.p.  $158^\circ$ .

*Acetyl*: pale yellow needles from MeOH. M.p.  $211^\circ$ .

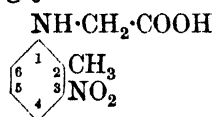
Lindemann, Krause, *J. prakt. Chem.*, 1927, **115**, 256.

Brady, Day, Reynolds, *J. Chem. Soc.*, 1929, 2265.

**6-Nitro-3 : 4-tolylenediamine** (6-Nitro-3 : 4-diaminotoluene).

4-N-*Me*:  $C_8H_{11}O_2N_3$ . MW, 181. Dark red needles from hot  $H_2O$  or EtOH.Aq. M.p.  $180^\circ$ .

See last reference above.

**3-Nitro-*o*-tolylglycine** $C_9H_{10}O_4N_2$ 

MW, 210

Yellowish-brown prisms from EtOH. M.p.  $152^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $PhNO_2$ . Spar. sol. hot  $H_2O$ ,  $Et_2O$ , AcOH. Insol.  $C_6H_6$ ,  $CHCl_3$ , pet. ether.

Pollak, *J. prakt. Chem.*, 1915, **91**, 297.

**4-Nitro-*o*-tolylglycine.**

Reddish-brown cryst. from EtOH. M.p.  $192^\circ$ . Very sol. hot  $H_2O$ . Sol. EtOH. Spar. sol.  $Me_2CO$ ,  $CHCl_3$ . Insol.  $Et_2O$ ,  $PhNO_2$ .

*Me ester*:  $C_{10}H_{12}O_4N_2$ . MW, 224. Yellow needles from  $C_6H_6$ . M.p.  $82^\circ$ . Sol. EtOH,  $C_6H_6$ .

*Et ester*:  $C_{11}H_{14}O_4N_2$ . MW, 238. Cryst. from  $C_6H_6$ . M.p.  $87^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ , pet. ether.

See previous reference.

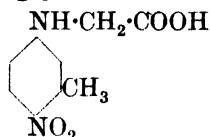
**5-Nitro-*o*-tolylglycine.**

Pale yellow needles from  $H_2O$ . M.p.  $140^\circ$ . Very sol.  $Me_2CO$ . Sol. EtOH,  $Et_2O$ , AcOH. Spar. sol.  $C_6H_6$ , cold  $H_2O$ .

*Me ester*: pale yellow needles from  $C_6H_6$ . M.p.  $108^\circ$ . Sol. EtOH, AcOH. Spar. sol.  $Et_2O$ . Insol.  $H_2O$ ,  $CHCl_3$ , pet. ether.

*Et ester*: reddish-brown needles from EtOH. M.p.  $42^\circ$ . Sol.  $Me_2CO$ . Mod. sol. EtOH,  $CHCl_3$ , AcOH. Spar. sol.  $C_6H_6$ . Insol.  $H_2O$ , pet. ether.

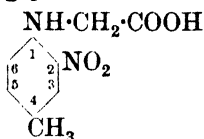
See previous reference.

4-Nitro-*m*-tolylglycine

$C_9H_{10}O_4N_2$  MW, 210

Yellow cryst. from  $H_2O$ . M.p.  $145^\circ$ . Sol. EtOH,  $Me_2CO$ ,  $PhNO_2$ . Spar. sol.  $Et_2O$ . Insol. cold  $H_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , pet. ether.

Pollak, *J. prakt. Chem.*, 1915, **91**, 304.

2-Nitro-*p*-tolylglycine

$C_9H_{10}O_4N_2$  MW, 210

Reddish-brown prisms from EtOH or AcOH. M.p.  $189-90^\circ$  decomp. Sol. hot EtOH, AcOH. Spar. sol. cold  $H_2O$ ,  $Et_2O$ .

*Et ester*:  $C_{11}H_{14}O_4N_2$ . MW, 238. Bright yellow needles from EtOH. M.p.  $65^\circ$ . Sol.  $C_6H_6$ , ligroin.

Plöchl, *Ber.*, 1886, **19**, 9.

Leuckart, Hermann, *Ber.*, 1887, **20**, 26.

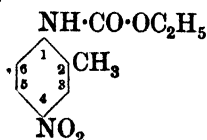
3-Nitro-*p*-tolylglycine.

Yellow prisms from EtOH. M.p.  $130^\circ$ . Very sol. EtOH, AcOH. Spar. sol.  $Et_2O$ ,  $PhNO_2$ . Insol. cold  $H_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

$NH_4$  salt: reddish-brown prisms. M.p.  $135^\circ$ . Sol.  $H_2O$ , EtOH,  $Et_2O$ , AcOH.

*Cu salt*: green cryst. +  $H_2O$ . M.p.  $160^\circ$ . Sol. EtOH. Spar. sol.  $Me_2CO$ . Insol. cold  $H_2O$ ,  $C_6H_6$ ,  $Et_2O$ ,  $CHCl_3$ .

Pollak, *J. prakt. Chem.*, 1915, **91**, 296.

4-Nitro-*o*-tolylurethane

$C_{10}H_{12}O_4N_2$

MW, 224

Pale yellow needles from EtOH. M.p.  $135^\circ$ . Sol. boiling  $H_2O$ . Mod. sol.  $C_6H_6$ ,  $Et_2O$ . Insol. boiling ligroin.

Vittenet, *Bull. soc. chim.*, 1899, **21**, 591.

Ryan, Cullinane, *Chem. Abstracts*, 1923, **17**, 1792.

5-Nitro-*o*-tolylurethane.

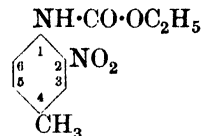
Needles from EtOH. M.p.  $137^\circ$  ( $129-30^\circ$ ). Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Mod. sol. hot  $H_2O$ . Spar. sol. boiling ligroin.

See last reference above and also Schiff, Vanni, *Ann.*, 1892, **268**, 323.

6-Nitro-*o*-tolylurethane.

Prisms. M.p.  $131^\circ$ . Spar. sol. ligroin.

Ryan, Cullinane, *Chem. Abstracts*, 1923, **17**, 1792.

2-Nitro-*p*-tolylurethane

$C_{10}H_{12}O_4N_2$  MW, 224

Yellow needles from EtOH. M.p.  $63^\circ$ . Sol.  $C_6H_6$ ,  $Et_2O$ . Mod. sol. boiling  $H_2O$ . Spar. sol. ligroin.

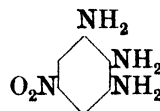
Vittenet, *Bull. soc. chim.*, 1899, **21**, 590.

3-Nitro-*p*-tolylurethane.

Pale yellow needles from EtOH.Aq. M.p.  $77-8^\circ$ . Mod. sol.  $C_6H_6$ . Spar. sol.  $Et_2O$ . Insol. boiling ligroin.

See previous reference.

## 5-Nitro-1 : 2 : 3-triaminobenzene



$C_6H_5O_2N_4$  MW, 168

Red cryst. with golden lustre from EtOH. Decomp. at  $260^\circ$ .

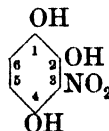
*Triacetyl deriv.*: m.p.  $243^\circ$ .

Nietzki, Hagenbach, *Ber.*, 1897, **30**, 543.

## Nitro-1 : 2 : 3-trihydroxybenzene.

See Nitropyrogallol.

3-Nitro-1 : 2 : 4-trihydroxybenzene (3-Nitrohydroxyhydroquinone)



MW, 171

$C_6H_5O_5N$

2-Me ether:  $C_7H_7O_5N$ . MW, 185. Red prisms from  $C_6H_6$ . M.p. 86.5–87.5°.

Dakin, *Am. Chem. J.*, 1909, **42**, 493.

**5-Nitro-1 : 2 : 4-trihydroxybenzene** (5-Nitrohydroxyhydroquinone).

Tri-Me ether:  $C_9H_{11}O_5N$ . MW, 213. Yellow needles from EtOH. M.p. 130°. Sol. boiling EtOH,  $C_6H_6$ , AcOH. Spar. sol. hot  $H_2O$ ,  $Et_2O$ . Conc.  $H_2SO_4 \rightarrow$  red sol.

Tri-Et ether:  $C_{12}H_{17}O_5N$ . MW, 255. Yellow needles from EtOH. M.p. 108–9°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , AcOH.

Brezina, *Monatsh.*, 1901, **22**, 347.

Fabinyi, Széki, *Ber.*, 1906, **39**, 3681.

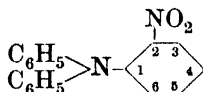
**Nitro-1 : 3 : 5-trihydroxybenzene.**

See Nitrophloroglucinol.

**Nitro-trimethoxyphthalide.**

See under 3-Nitro-opianic Acid.

**2-Nitrotriphenylamine**



$C_{18}H_{14}O_2N_2$  MW, 290

Yellowish-orange cryst. from EtOH. M.p. 98°.

Piccard, Larsen, *J. Am. Chem. Soc.*, 1917, **39**, 2009.

**3-Nitrotriphenylamine.**

Yellow cryst. from MeOH. M.p. 78°. Sol.  $Et_2O$ ,  $C_6H_6$ , AcOEt,  $PhNO_2$ . Mod. sol. EtOH, AcOH. Insol.  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  colourless sol. turning blue in few seconds.

See previous reference.

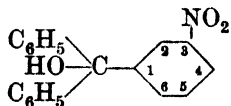
**4-Nitrotriphenylamine.**

Golden-yellow plates from AcOH–AcOEt. M.p. 144° (140°). Very sol.  $CHCl_3$ . Sol.  $Me_2CO$ ,  $C_6H_6$ ,  $CS_2$ , AcOEt, hot AcOH. Spar. sol. EtOH, ligroin. Insol. cold AcOH.

Gambarjan, *Ber.*, 1908, **41**, 3510.

Kawai, *J. Chem. Soc. Japan*, 1928, **49**, 235.

**3-Nitrotriphenylcarbinol** (3-Nitro- $\alpha$ -hydroxytriphenylmethane, 3-nitrotritanol)



$C_{19}H_{15}O_3N$  MW, 305

Cryst. from ligroin or AcOH.Aq. M.p. 75°.

Tsacher, *Ber.*, 1888, **21**, 190.

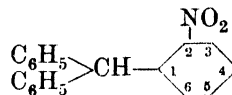
Kovachi, *Ann. chim.*, 1918, **10**, 202.

**4-Nitrotriphenylcarbinol** (4-Nitro- $\alpha$ -hydroxytriphenylmethane, 4-nitrotritanol).

Prisms from ligroin. M.p. 97–8°.

Baeyer, Villiger, *Ber.*, 1904, **37**, 606.

**2-Nitrotriphenylmethane** (2-Nitrotritanol)



$C_{19}H_{15}O_2N$  MW, 289

Pale yellowish leaflets from EtOH or MeOH. M.p. 93–4°. Sol. hot EtOH, ligroin. Mod. sol. AcOH.

Kliegl, *Ber.*, 1907, **40**, 4941.

**3-Nitrotriphenylmethane** (3-Nitrotritanol).

Cryst. from ligroin or  $Et_2O$ . M.p. 90°.

Tsacher, *Ber.*, 1888, **21**, 188.

Kovache, *Ann. chim.*, 1918, **10**, 202.

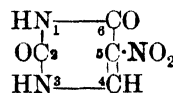
**4-Nitrotriphenylmethane** (4-Nitrotritanol).

Leaflets from EtOH. M.p. 93°.

Baeyer, Löhr, *Ber.*, 1890, **23**, 1622.

Stolz, *D.R.P.*, 40,340.

**5-Nitrouracil**



$C_4H_3O_4N_3$  MW, 157

Golden-yellow needles. Decomp. on heating. Spar. sol.  $H_2O$ , cold EtOH. Reacts as monobasic acid.  $KMnO_4 \rightarrow CO_2 +$  oxalic acid + urea.

3-N-Me:  $C_5H_5O_4N_3$ . MW, 171. Long needles from EtOH. M.p. 255–6°. Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Reacts acid.

1 : 3-N-Di-Me:  $C_6H_7O_4N_3$ . MW, 185. Long needles from  $H_2O$ . M.p. 154.5°.

3-N-Et:  $C_6H_7O_4N_3$ . MW, 185. Long needles from  $H_2O$ . M.p. 194.5°.

1-N-Me-3-N-Et:  $C_7H_9O_4N_3$ . MW, 199. Pearly cryst. from  $H_2O$ . M.p. 73°. Sol. EtOH,  $Et_2O$ .

3-N-Me-1-N-Et: needles from  $H_2O$ . M.p. 109°.

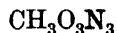
Picrate: m.p. 247°.

Johnson, Matsuo, *J. Am. Chem. Soc.*, 1919, **41**, 783.

Biltz, Heyn, *Ann.*, 1917, **413**, 110.

Levene, La Forge, *Ber.*, 1912, **45**, 618.

## Nitrourea



MW, 105

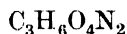
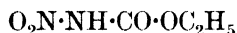
Leaflets or prisms from EtOH. M.p. 159° (150°) decomp. Very sol. Me<sub>2</sub>CO, AcOH. Sol. EtOH. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether.  $k = 7.0 \times 10^{-3}$  at 20°. Very stable towards ox. agents. Can be detonated. Not sensitive to percussion or heating.

Backer, *Rec. trav. chim.*, 1912, **31**, 22.

Willstätter, Pfannenstiel, *Ber.*, 1926, **59**, 1870.

Ingersoll, Armendt, *Organic Syntheses*, Collective Vol. I, 408.

## Nitrourethane

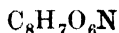
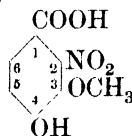


MW, 134

Plates from Et<sub>2</sub>O or ligroin. M.p. 64°. Very sol. EtOH, Et<sub>2</sub>O. Sol. H<sub>2</sub>O. Spar. sol. ligroin.  $k = 4.83 \times 10^{-4}$  at 20°. Decomp. at 140° or by heating with H<sub>2</sub>O.

Thiele, Lachmann, *Ann.*, 1895, **288**, 287.

**2-Nitrovanillic Acid** (2-Nitroprotocatechuic acid 3-methyl ether)



MW, 213

Needles from EtOH.Aq., cryst. from C<sub>6</sub>H<sub>6</sub>. Decomp. at 246° (210°). Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.  $k = 1.2 \times 10^{-4}$  at 25°.

4-Me ether: see 2-Nitroveratric Acid.

Acetyl: needles from EtOH.Aq. M.p. 181-2°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. hot H<sub>2</sub>O.

Tiemann, Matsmoto, *Ber.*, 1876, **9**, 943.

Klemenc, *Monatsh.*, 1914, **35**, 94.

**5-Nitrovanillic Acid** (5-Nitroprotocatechuic acid 3-methyl ether).

Pale yellow cryst. from H<sub>2</sub>O. M.p. 216°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Spar. sol. H<sub>2</sub>O, CHCl<sub>3</sub>. NH<sub>3</sub>.Aq. → intense orange-yellow col.

4-Me ether: see 5-Nitroveratric Acid.

Me ester: C<sub>9</sub>H<sub>7</sub>O<sub>6</sub>N. MW, 227. Yellow needles from MeOH or leaflets from Et<sub>2</sub>O. M.p. 155° (148-9°). Sol. C<sub>6</sub>H<sub>6</sub>, EtOH. Less sol. ligroin, Et<sub>2</sub>O. Sol. alkalis with red col. Benzoyl: yellowish-brown needles from EtOH. M.p. 124-5°. Sol. C<sub>6</sub>H<sub>6</sub>, warm Et<sub>2</sub>O, AcOH, ligroin.

Chloride: C<sub>8</sub>H<sub>6</sub>O<sub>5</sub>NCl. MW, 231.5. M.p.

93-4°. Sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O, ligroin. Insol. pet. ether.

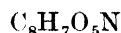
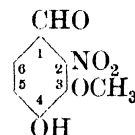
Nitrile: C<sub>8</sub>H<sub>6</sub>O<sub>4</sub>N<sub>2</sub>. MW, 194. Needles from H<sub>2</sub>O. M.p. 140°. Acetyl: yellowish needles from EtOH.Aq. M.p. 102°.

Borsche, *Ber.*, 1917, **50**, 1346.

v. Konek, Pacsu, *Ber.*, 1918, **51**, 861.

Klemenc, *Monatsh.*, 1912, **33**, 388; 1914, **35**, 94.

**2-Nitrovanillin** (2-Nitroprotocatechuic aldehyde 3-methyl ether)



MW, 197

Needles from EtOH. M.p. 137°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Less sol. hot H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. ligroin, pet. ether. Turns brown in air.

Me ether: see 2-Nitroveratric Aldehyde.

Et ether: C<sub>10</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 225. Cryst. from EtOH. M.p. 106-7°.

Acetyl: needles from EtOH.Aq., prisms from ligroin. M.p. 85-7°. Spar. sol. H<sub>2</sub>O, ligroin, pet. ether. Turns yellow in air. Phenylhydrazone: reddish-brown plates. M.p. 154°.

Phenylhydrazone: prisms from AcOH. M.p. 161-2°.

Slotta, Lauersen, *J. prakt. Chem.*, 1934, **139**, 224.

Pschorr, Sumuleanu, *Ber.*, 1899, **32**, 3407.

Raiford, Stoesser, *J. Am. Chem. Soc.*, 1928, **50**, 2559.

Späth, Tharrer, *Ber.*, 1933, **66**, 911.

**5-Nitrovanillin** (5-Nitroprotocatechuic aldehyde 3-methyl ether).

Pale yellow plates from AcOH. M.p. 178° (172°). Very sol. hot AcOH. Sol. CHCl<sub>3</sub>, CS<sub>2</sub>. Spar. sol. hot EtOH. Insol. Et<sub>2</sub>O.

Me ether: see 5-Nitroveratric Aldehyde.

Acetyl: yellow cryst. from AcOEt. M.p. 88°. Sol. AcOH, AcOEt.

Oxime: pale orange-yellow needles from EtOH, deep orange-yellow needles from AcOH. M.p. 216° (200-1°). Sol. EtOH, Et<sub>2</sub>O. Spar. sol. hot H<sub>2</sub>O. Acetyl: orange needles + H<sub>2</sub>O from EtOH.Aq. M.p. 147°. Diacetyl: yellow cryst. from EtOH. M.p. 112°. B.HCl: m.p. 204° decomp.

Vogl, *Monatsh.*, 1899, **20**, 384.

Brady, Dunn, *J. Chem. Soc.*, 1915, **107**, 1861.

**6-Nitrovanillin** (6-Nitroprotocatechuic aldehyde 3-methyl ether).

Yellow plates from AcOH. M.p. 212° (207°). Sol. hot H<sub>2</sub>O, most org. solvents.

*Me ether*: see 6-Nitroveratric Aldehyde.

*Et ether*: cryst. M.p. 159–60°. Sublimes.

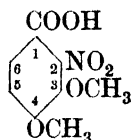
*p-Nitrobenzyl ether*: pale yellow needles from AcOEt. M.p. 212–14°. Sol. Me<sub>2</sub>CO, Py, hot C<sub>6</sub>H<sub>6</sub>, AcOH, AcOEt. Spar. sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. *Phenylhydrazone*: brick-red needles from AcOH or EtOH.Aq. M.p. 208°. *Oxime*: pale yellow needles from EtOH. M.p. 158–60°. *Anil*: yellow prisms from EtOH. M.p. 192–3°.

*Phenylhydrazone*: dark red needles from C<sub>6</sub>H<sub>6</sub>. M.p. 198°.

Raiford, Stoesser, *J. Am. Chem. Soc.*, 1928, **50**, 2559.

Nair, Robinson, *J. Chem. Soc.*, 1932, 1237.

**2-Nitroveratric Acid** (2-Nitroprotocatechuic acid dimethyl ether, 2-nitro-3:4-dimethoxybenzoic acid)



C<sub>9</sub>H<sub>9</sub>O<sub>6</sub>N

MW, 227

Needles from H<sub>2</sub>O. M.p. 203°. Sol. most ord. solvents. Spar. sol. C<sub>6</sub>H<sub>6</sub>, pet. ether. Sol. 250 parts boiling, 1000 parts cold H<sub>2</sub>O.

*Me ester*: C<sub>10</sub>H<sub>11</sub>O<sub>6</sub>N. MW, 241. Needles from EtOH.Aq. M.p. 127–8°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

*Chloride*: C<sub>9</sub>H<sub>8</sub>O<sub>5</sub>NCl. MW, 245.5. Needles from toluene. M.p. 78° (73°). Sol. CS<sub>2</sub>, toluene, xylene. Spar. sol. C<sub>6</sub>H<sub>6</sub>, Et<sub>2</sub>O. Insol. ligroin. Decomp. by H<sub>2</sub>O.

*Amide*: C<sub>9</sub>H<sub>10</sub>O<sub>5</sub>N<sub>2</sub>. MW, 226. Needles from toluene, plates from H<sub>2</sub>O. M.p. 172°. Sol. toluene, xylene. Spar. sol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOEt, CS<sub>2</sub>.

Pisovschi, *Ber.*, 1910, **43**, 2140.

Pschorr, Sumuleanu, *Ber.*, 1899, **32**, 3410.

### 5-Nitroveratric Acid.

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 196° (194°). Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, CHCl<sub>3</sub>, cold C<sub>6</sub>H<sub>6</sub>. Very spar. sol. pet. ether.

*Me ester*: needles from MeOH or pet. ether. M.p. 78°. Sol. usual solvents. Mod. sol. MeOH, pet. ether.

Simonsen, Rau, *J. Chem. Soc.*, 1918, **113**, 24.

Klemenc, *Monatsh.*, 1914, **35**, 96.

Zincke, Francke, *Ann.*, 1896, **293**, 192.

### 6-Nitroveratric Acid.

Yellow needles +  $\frac{1}{2}$  H<sub>2</sub>O. M.p. 185–7° (192–3°). Sol. 25 parts hot H<sub>2</sub>O. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.  $k = 3.6 \times 10^{-3}$  at 25°.

*Me ester*: needles. M.p. 143–4°. Sol. Et<sub>2</sub>O, boiling EtOH. Spar. sol. cold H<sub>2</sub>O.

*Et ester*: prisms from EtOH.Aq. M.p. 99–100°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Nitrile*: yellow needles from EtOH. M.p. 165°. Sol. CHCl<sub>3</sub>. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Conc. H<sub>2</sub>SO<sub>4</sub> → green sol. rapidly turning brown.

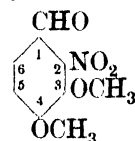
Keffler, *J. Chem. Soc.*, 1921, **119**, 1479.

Tiemann, Matsumoto, *Ber.*, 1876, **9**, 938.

Simonsen, Rau, *J. Chem. Soc.*, 1918, **113**, 26.

Pschorr, Sumuleanu, *Ber.*, 1899, **32**, 3410.

**2-Nitroveratric Aldehyde** (2-Nitroprotocatechuic aldehyde dimethyl ether, 2-nitro-3:4-dimethoxybenzaldehyde)



C<sub>9</sub>H<sub>9</sub>O<sub>5</sub>N

MW, 211

Prisms from EtOH.Aq. M.p. 64°. Spar. sol. H<sub>2</sub>O. Insol. ligroin, pet. ether. Sol. most other solvents. Turns yellow in air.

*Phenylhydrazone*: yellow plates from AcOH. M.p. 194°.

Pschorr, Stöhrer, *Ber.*, 1902, **35**, 4397.

Pschorr, *Ber.*, 1906, **39**, 3108.

Slota, Lauersen, *J. prakt. Chem.*, 1934, **139**, 225.

### 5-Nitroveratric Aldehyde.

Needles from EtOH.Aq. M.p. 90–1°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, pet. ether. *anti-Oxime*: plates from EtOH. M.p. 151°. *Acetyl*: plates from Me<sub>2</sub>CO.Aq. M.p. 115°.

*Phenylhydrazone*: yellow leaflets. M.p. 108–10°.

Pschorr, Stöhrer, *Ber.*, 1902, **35**, 4399.

Brady, Manjunath, *J. Chem. Soc.*, 1924, **125**, 1067.

### 6-Nitroveratric Aldehyde.

Yellow needles from EtOH. M.p. 133.5–134.5°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, AcOH, hot EtOH. Spar. sol. H<sub>2</sub>O, ligroin.

*Di-Me acetal*: prisms from Et<sub>2</sub>O–pet. ether. M.p. 54.5–55.5°. Sol. most org. solvents.

*anti-Oxime*: yellow needles from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 178–80°. Sol. hot EtOH. Mod.

sol. cold EtOH, Et<sub>2</sub>O, hot C<sub>6</sub>H<sub>6</sub>. *Acetyl*: pale yellow leaflets from EtOH. M.p. 152°.

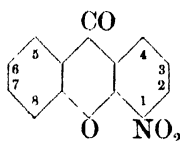
*Phenylhydrazone*: plates from AcOH. M.p. 216-18°.

See previous references and also  
 Pschorr, Sumuleanu, *Ber.*, 1899, **32**, 3412.  
 Marr, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 1329.

#### Nitroveratrol.

See under Nitrocatechol.

#### 1-Nitroxanthone



C<sub>13</sub>H<sub>7</sub>O<sub>4</sub>N MW, 241

Yellow needles from EtOH. M.p. 127°.  
 H<sub>2</sub>SO<sub>4</sub> → green sol.

Dhar, *J. Chem. Soc.*, 1920, **117**, 1063.

#### 2-Nitroxanthone.

Pale yellowish needles from EtOH.Aq. M.p. 176°. Sol. C<sub>6</sub>H<sub>6</sub>, boiling EtOH. Spar. sol. ligroin. Insol. H<sub>2</sub>O. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with strong blue fluor.

See previous reference and also  
 Ullmann, Wagner, *Ann.*, 1907, **355**, 362.

#### 3-Nitroxanthone.

Brown needles from EtOH. M.p. 200°. Insol. usual solvents. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with greenish-blue fluor.

Dhar, *J. Chem. Soc.*, 1920, **117**, 1062.  
 Purgotti, *Gazz. chim. ital.*, 1914, **44**, i, 643.

#### 4-Nitroxanthone.

Brown needles from EtOH. M.p. 210°. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

Dhar, *J. Chem. Soc.*, 1920, **117**, 1061.

#### 3-Nitro-*o*-xylene



C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N MW, 151

Needles from EtOH. M.p. 15°. B.p. 240°/760 mm., 136°/29 mm., 131°/20 mm.

Crossley, Wren, *J. Chem. Soc.*, 1911, **99**, 2342.

Crossley, Renouf, *J. Chem. Soc.*, 1909, **95**, 208.

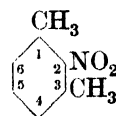
#### 4-Nitro-*o*-xylene.

Yellow prisms from EtOH. M.p. 30°. B.p. 254°/748 mm., 143°/21 mm. Misc. with EtOH above 30°. Sol. most org. solvents.

Diepolder, *Ber.*, 1909, **42**, 2918.

Crossley, Renouf, *J. Chem. Soc.*, 1909, **95**, 207, 215.

#### 2-Nitro-*m*-xylene



C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N MW, 151

Liq. B.p. 225°/744 mm. D<sup>15</sup> 1.112.

Grevingk, *Ber.*, 1884, **17**, 2430.

Miolati, Lotti, *Gazz. chim. ital.*, 1897, **27**, i, 297.

#### 4-Nitro-*m*-xylene.

M.p. 2°. B.p. 237-9° (245.5°/744 mm.). D<sup>1</sup> 1.135, D<sup>17.5</sup> 1.126.

Grevingk, *Ber.*, 1884, **17**, 2429.

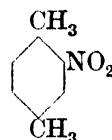
Harmsen, *Ber.*, 1880, **13**, 1558.

#### 5-Nitro-*m*-xylene.

Needles from EtOH. M.p. 75°. B.p. 273°/739 mm.

Noyes, *Am. Chem. J.*, 1898, **20**, 800.

#### 2-Nitro-*p*-xylene



C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N MW, 151

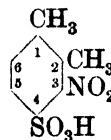
Pale yellowish liq. B.p. 234-7°. D<sup>15</sup> 1.132.

Jannasch, *Ann.*, 1875, **176**, 55.

#### ω-Nitroxylene.

See Tolylnitromethane.

#### 3-Nitro-*o*-xylene-4-sulphonic Acid



C<sub>8</sub>H<sub>9</sub>O<sub>5</sub>NS MW, 231

Needles + 3H<sub>2</sub>O.

*Amide*: C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>N<sub>2</sub>S. MW, 230. Prismatic needles from EtOH. M.p. 214°. Spar. sol. EtOH.

Simonsen, *J. Chem. Soc.*, 1913, **103**, 1148.



**5-Nitro-*o*-xylene-4-sulphonic Acid.**

Needles. Hygroscopic.

*Amide*: prismatic needles from EtOH. M.p. 157-8°.

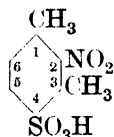
See previous reference.

**6-Nitro-*o*-xylene-4-sulphonic Acid.**

Needles. Easily decomp.

*Chloride*:  $C_8H_8O_4NClS$ . MW, 249.5. Plates from  $C_6H_6$ -pet. ether. M.p. 69-70°.*Amide*: needles from EtOH.Aq. M.p. 180°.

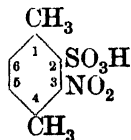
See previous reference.

**2-Nitro-*m*-xylene-4-sulphonic Acid** $C_8H_8O_5NS$  MW, 231Leaflets +  $H_2O$  from  $H_2O$ . M.p. anhyd. 144°.*Chloride*:  $C_8H_8O_4NClS$ . MW, 249.5. Cryst. M.p. 96°.*Amide*:  $C_8H_{10}O_4N_2S$ . MW, 230. Needles. M.p. 172°. Sol. EtOH,  $Et_2O$ .Claus, Schmidt, *Ber.*, 1886, 19, 1418.**5-Nitro-*m*-xylene-4-sulphonic Acid.**Leaflets from  $HNO_3$ . M.p. 95-100°. Hygroscopic. Sol.  $H_2O$ .*Chloride*: plates. M.p. 97°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .*Amide*: needles from EtOH.Aq. M.p. 108°.

See previous reference.

**6-Nitro-*m*-xylene-4-sulphonic Acid.**Needles from dil.  $HNO_3$ . M.p. 132° (122°). Sol. dil.  $HNO_3$ .*Fluoride*:  $C_8H_8O_4NF_3S$ . MW, 233. Cryst. from  $C_6H_6$ -pet. ether. M.p. 109-10°.*Chloride*: cryst. M.p. 98°.*Amide*: needles. M.p. 187° (179°).Limpricht, v. Riesen, *Ber.*, 1885, 18, 2191.Steinkopf *et al.*, *J. prakt. Chem.*, 1927, 117, 41.

See also previous reference.

**3-Nitro-*p*-xylene-2-sulphonic Acid** $C_8H_8O_5NS$  MW, 231Plates +  $H_2O$ . M.p. anhyd. 143-5° part. decomp. Mod. sol. EtOH. Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , pet. ether.*Phenyl ester*:  $C_{14}H_{13}O_5NS$ . MW, 307. Prisms from EtOH. M.p. 83-83.5°.*o-Tolyl ester*:  $C_{15}H_{15}O_5NS$ . MW, 321. Prisms from EtOH. M.p. 151.5-152°.*m-Tolyl ester*: plates from EtOH. M.p. 107.5-108°.*p-Tolyl ester*: plates from EtOH. M.p. 76-7°.*Chloride*:  $C_8H_8O_4NClS$ . MW, 249.5. Prisms from  $Et_2O$ -pet. ether. M.p. 109.5-110.5°.*Amide*:  $C_8H_{10}O_4N_2S$ . MW, 230. Needles from 50% EtOH. M.p. 191-2°.*Anilide*:  $C_{14}H_{14}O_4N_2S$ . MW, 306. Bluish needles from EtOH.Aq. M.p. 181.5-182.5°. Sol. most org. solvents. Insol.  $H_2O$ .*o-Toluidide*:  $C_{15}H_{16}O_4N_2S$ . MW, 320. Plates from 50% EtOH. M.p. 143.5-145°.*p-Toluidide*: needles from EtOH. M.p. 158.5-159°. Sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ . Less sol.  $Et_2O$ , pet. ether. Insol.  $H_2O$ .Karslake, Huston, *J. Am. Chem. Soc.*, 1914, 36, 1253.Huston, *J. Am. Chem. Soc.*, 1915, 37, 2120.**5-Nitro-*p*-xylene-2-sulphonic Acid.**Plates +  $H_2O$  from HCl. M.p. 138-40°. Very hygroscopic. Bitter taste.*Phenyl ester*: yellowish plates from EtOH. M.p. 120-120.5°.*o-Tolyl ester*: needles from EtOH. M.p. 99-100°.*m-Tolyl ester*: needles from EtOH. M.p. 110-11°.*p-Tolyl ester*: plates from EtOH. M.p. 117.5-118.5°.*Chloride*: plates from  $Et_2O$  or  $Et_2O$ -pet. ether. M.p. 74.5-75.5°.*Amide*: prismatic plates from 50% EtOH. M.p. 197-8°.*Anilide*: plates from EtOH. M.p. 130.5-131°.*o-Toluidide*: yellowish plates from 50% EtOH. M.p. 140.5-141°.*p-Toluidide*: plates from EtOH. M.p. 143.5-144.5°.

See previous references and also

Choufoer, *Chem. Abstracts*, 1925, 19, 2195.**6-Nitro-*p*-xylene-2-sulphonic Acid.**Plates +  $H_2O$ . Decomp. at 128°. Sol. EtOH. Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ ,  $CS_2$ . Very hygroscopic. Bitter taste.*Phenyl ester*: plates from EtOH. M.p. 117-18°. Mod. sol.  $CHCl_3$ ,  $CCl_4$ ,  $C_6H_6$ . Insol.  $H_2O$ .*o-Tolyl ester*: cryst. from EtOH. M.p. 66-7°. Mod. sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. pet. ether. Insol.  $H_2O$ .

*m*-Tolyl ester: plates from EtOH. M.p. 71.5–72°. Less sol. than *o*-tolyl ester.

*p*-Tolyl ester: plates from EtOH. M.p. 93.5–94.5°. Less sol. than *m*-tolyl ester.

Fluoride:  $C_8H_8O_4NFS$ . MW, 233. Cryst. from  $C_6H_6$ -pet. ether. M.p. 74–74.5°.

Chloride: prisms from  $Et_2O$ -pet. ether. M.p. 61°. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ ,  $CS_2$ . Spar. sol. pet. ether.

Amide: plates from EtOH.Aq. M.p. 172–3°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $H_2O$ .

Anilide: needles from EtOH.Aq. M.p. 143–4°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $H_2O$ .

*o*-Toluidide: needles from 50% EtOH. M.p. 126.5–127.5°. Sol. EtOH,  $CHCl_3$ ,  $CCl_4$ ,  $C_6H_6$ . Less sol.  $Et_2O$ , pet. ether. Insol.  $H_2O$ .

*p*-Toluidide: cryst. from EtOH.Aq. M.p. 135–6°.

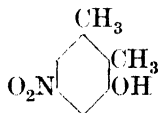
Karslake, *J. Am. Chem. Soc.*, 1914, **36**, 1251.

Huston, *J. Am. Chem. Soc.*, 1915, **37**, 2119.

Choufoer, *Chem. Abstracts*, 1925, **19**, 2195.

Steinkopf *et al.*, 1927, **117**, 40.

### 5-Nitro-*o*-3-xenol



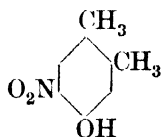
$C_8H_9O_3N$

MW, 167

Orange-yellow needles from  $C_6H_6$  or  $CHCl_3$ . M.p. 109°.

Crossley, *J. Chem. Soc.*, 1913, **103**, 2180.

### 5-Nitro-*o*-4-xenol



$C_8H_9O_3N$

MW, 167

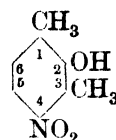
Yellow rhombic cryst. from EtOH. M.p. 87°. Very sol.  $CHCl_3$ . Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. pet. ether. Volatile in steam.

*Me ether*:  $C_9H_{11}O_3N$ . MW, 181. Pale yellow cryst. from EtOH. M.p. 79°.

Diepolder, *Ber.*, 1909, **42**, 2917.

Cain, Simonsen, *J. Chem. Soc.*, 1914, **105**, 163.

### 4-Nitro-*m*-2-xenol



$C_8H_9O_3N$

MW, 167

Leaflets and prisms from  $C_6H_6$ , needles from ligroin. M.p. 99–100°.

Auwers, Markovits, *Ber.*, 1908, **41**, 2338.

### 5-Nitro-*m*-2-xenol.

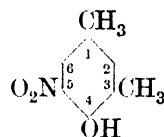
Prisms from MeOH. M.p. 169–70°. Very sol.  $CHCl_3$ . Sol. EtOH. Spar. sol.  $C_6H_6$ , ligroin.

*Me ether*:  $C_9H_{11}O_3N$ . MW, 181. Pale yellow needles from EtOH. M.p. 92°. Pleasant odour.

Auwers, Markovits, *Ber.*, 1908, **41**, 2336.

Rowe, Bannister, Story, *J. Soc. Chem. Ind.*, 1931, **50**, 79r.

### 5-Nitro-*m*-4-xenol



$C_8H_9O_3N$

MW, 167

Yellow needles from EtOH. M.p. 78° (72°). Volatile in steam.

*Me ether*:  $C_9H_{11}O_3N$ . MW, 181. Prisms from  $Et_2O$ . M.p. 27°. B.p. 269.5°.

*p*-Toluenesulphonyl: plates from EtOH. M.p. 111–12°.

Hodgkinson, Limpach, *J. Chem. Soc.*, 1893, **63**, 105.

Fries, Kann, *Ann.*, 1907, **353**, 354.

Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2042.

Fox, Turner, *J. Chem. Soc.*, 1930, 1866.

### 6-Nitro-*m*-4-xenol.

Yellow needles from HCl. M.p. 95°.

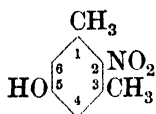
*Me ether*: needles from EtOH. M.p. 56–7°. Mod. sol. EtOH,  $Et_2O$ .

Pfaff, *Ber.*, 1883, **16**, 616.

Bamberger, Reber, *Ber.*, 1907, **40**, 2267.

Maltese, *Gazz. chim. ital.*, 1907, **37**, ii, 284.

Manske, *Organic Syntheses*, Collective Vol. I, 397, Note 5.

2-Nitro-*m*-5-xlenol $C_8H_9O_3N$ 

MW, 167

Yellowish prisms from ligroin. M.p. 107–8°. Non-volatile in steam.

*Me ether*:  $C_9H_{11}O_3N$ . MW, 181. Needles from EtOH.Aq. M.p. 53°.

Auwers, Borsche, *Ber.*, 1915, **48**, 1715.

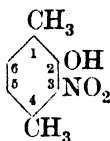
Rowe, Bannister, Seth, Storey, *J. Soc. Chem. Ind.*, 1930, **49**, 469t.

4-Nitro-*m*-5-xlenol.

Yellow needles from ligroin or MeOH.Aq. M.p. 66°. Sol. most org. solvents. Spar. sol. ligroin, pet. ether. Volatile in steam.

*Me ether*: rhombic plates from MeOH. M.p. 45–6°. Volatile in steam.

See previous references.

3-Nitro-*p*-2-xlenol $C_8H_9O_3N$ 

MW, 167

Golden-yellow needles from pet. ether. M.p. 34–5°. B.p. 236° decomp., 150°/15 mm. Sol. most org. solvents.

*Benzoyl*: m.p. 79–80°.

Auwers, Michaelis, *Ber.*, 1914, **47**, 1289, Note 4.

Oliveri, *Gazz. chim. ital.*, 1882, **12**, 163.

5-Nitro-*p*-2-xlenol.

Pale yellow needles from EtOH.Aq. M.p. 121–3° (115°). Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O.

*Et ether*:  $C_{10}H_{13}O_3N$ . MW, 195. M.p. 85°.

*Acetyl*: yellow prisms from EtOH. M.p. 72–3°. Sol. CHCl<sub>3</sub>, hot EtOH.

Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2042.

Noelting, *Ber.*, 1904, **37**, 2593.

See also previous references.

6-Nitro-*p*-2-xlenol.

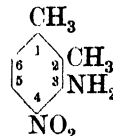
Yellow leaflets from pet. ether. M.p. 91°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Volatile in steam.

*Me ether*:  $C_9H_{11}O_3N$ . MW, 181. Needles

from MeOH. M.p. 62–62.5°. Sol. most org. solvents.

Sonn, *Ber.*, 1916, **49**, 2589.

Kostanecki, *Ber.*, 1886, **19**, 2321.

4-Nitro-*o*-3-xyldine $C_8H_{10}O_2N_2$ 

MW, 166

Red plates from EtOH. M.p. 118–19°. Sol. very conc. HCl.

*Acetyl*: needles from EtOH. M.p. 160°.

*Benzoyl*: cryst. M.p. 177–8°. Mod. sol. EtOH.

Noelting, Braun, Thesmar, *Ber.*, 1901, **34**, 2246.

5-Nitro-*o*-3-xyldine.

Pale yellow needles from EtOH. M.p. 111–12°.

*Acetyl*: needles from EtOH. M.p. 230–1°.

*Benzoyl*: m.p. 227–8°. Mod. sol. EtOH.

Crossley, Morrell, *J. Chem. Soc.*, 1911, **99**, 2351.

See also previous reference.

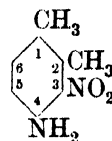
6-Nitro-*o*-3-xyldine.

Brownish-yellow prisms from EtOH. M.p. 114°.

*Acetyl*: yellowish needles from EtOH. M.p. 149–50°.

*Benzoyl*: yellowish cryst. M.p. 208–9°. Mod. sol. EtOH.

Noelting, Braun, Thesmar, *Ber.*, 1901, **34**, 2245.

3-Nitro-*o*-4-xyldine $C_8H_{10}O_2N_2$ 

MW, 166

Red prisms from EtOH. M.p. 65–6°. Volatile in steam.

*Acetyl*: needles from EtOH. M.p. 115–16°.

*Benzoyl*: needles. M.p. 199–200°. Spar. sol. EtOH.

Noelting, Braun, Thesmar, *Ber.*, 1901, **34**, 2249.

Crossley, Wren, *J. Chem. Soc.*, 1911, **99**, 2342.

**5-Nitro-*o*-4-xylylidine.**

Brownish-red prisms from EtOH. M.p. 139–40°. Mod. sol. org. solvents. Sol. about 40 parts Et<sub>2</sub>O.

*Acetyl*: pale yellow needles from EtOH. M.p. 107°.

*Benzoyl*: pale yellow needles from EtOH. M.p. 149–50°.

Noelting, Braun, Thesmar, *Ber.*, 1901, **34**, 2248.

Diepolder, *Ber.*, 1909, **42**, 2917.

**6-Nitro-*o*-4-xylylidine.**

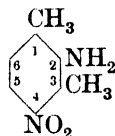
Orange leaflets from EtOH. M.p. 74–5°. Volatile in steam.

*Acetyl*: needles from EtOH. M.p. 209–10°.

*Benzoyl*: needles from EtOH. M.p. 223–4°.

Noelting, Braun, Thesmar, *Ber.*, 1901, **34**, 2250.

Noelting, Thesmar, *Ber.*, 1902, **35**, 632.

**4-Nitro-*m*-2-xylylidine**

C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

MW, 166

Yellow needles from EtOH.Aq. M.p. 81–2°.

*Acetyl*: pale yellowish needles. M.p. 170°.

Noelting, Stocklin, *Ber.*, 1891, **24**, 568.

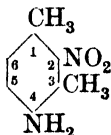
Noelting, Braun, Thesmar, *Ber.*, 1901, **34**, 2259.

**5-Nitro-*m*-2-xylylidine.**

Orange-yellow needles. M.p. 158°.

*N-Acetyl*: m.p. 178°.

Ibbotson, Kenner, *J. Chem. Soc.*, 1923, **123**, 1267.

**2-Nitro-*m*-4-xylylidine**

C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>

MW, 166

Golden-yellow needles. M.p. 81–2° (78°). Sol. EtOH, ligroin, hot H<sub>2</sub>O.

*Acetyl*: needles. M.p. 149°.

*Benzoyl*: needles from EtOH. M.p. 236°. Spar. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

Grevingk, *Ber.*, 1884, **17**, 2425.

Blanksma, *Rec. trav. chim.*, 1909, **28**, 94.

Noelting, Braun, Thesmar, *Ber.*, 1901, **34**, 2260.

**5-Nitro-*m*-4-xylylidine.**

Orange-red needles or plates from ligroin. M.p. 76° (70°).

*N-Me*: C<sub>9</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 180. Red plates with green reflex. M.p. 58°. Very sol. most solvents. Mod. sol. EtOH, ligroin.

*Acetyl*: yellowish needles from H<sub>2</sub>O. M.p. 172–3°.

*Benzoyl*: needles from EtOH. M.p. 184–5°.

*p-Nitrobenzoyl*: cryst. from AcOH. M.p. 139–40°. Sol. AcOH. Spar. sol. EtOH.

*p-Toluy*: yellowish needles from EtOH. M.p. 187°. Sol. AcOH. Mod. sol. hot EtOH. Insol. H<sub>2</sub>O.

Pinnow, Oesterreich, *Ber.*, 1898, **31**, 2931.

Willgerodt, Schmierer, *Ber.*, 1905, **38**, 1473.

Blanksma, *Rec. trav. chim.*, 1906, **25**, 181.

Hübner, *Ann.*, 1881, **208**, 320.

Ibbotson, Kenner, *J. Chem. Soc.*, 1923, **123**, 1268.

**6-Nitro-*m*-4-xylylidine.**

Orange-red needles. M.p. 123°. Sol. boiling EtOH.

*N-Me*: *p-toluenesulphonyl*, prisms from EtOH. M.p. 135–6°. *Benzenesulphonyl*: prisms from EtOH. M.p. 185–6°.

*N-Di-Me*: C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub>. MW, 194. Oil. *Picrate*: cryst. M.p. 170–3° decomp.

*Acetyl*: needles. M.p. 160°. Sol. EtOH. Mod. sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

*Diacetyl*: plates. M.p. 115°. Very sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH. Insol. H<sub>2</sub>O.

*Benzoyl*: needles from EtOH. M.p. 200°. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, pct. ether.

*Benzenesulphonyl*: prisms from EtOH. M.p. 148–5°.

*p-Toluenesulphonyl*: pale yellow cryst. from EtOH. M.p. 192°.

Noelting, Collin, *Ber.*, 1884, **17**, 265.

Errera, Maltese, *Gazz. chim. ital.*, 1903, **33**, ii, 283.

Fittig, Ahrens, Mattheides, *Ann.*, 1868, **147**, 18.

**2-Nitro-*m*-5-xylylidine.**

Orange prisms from ligroin. M.p. 132°.

*Acetyl*: prisms from EtOH.Aq. M.p. 163°.

Ibbotson, Kenner, *J. Chem. Soc.*, 1923, **123**, 1267.

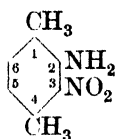
**4-Nitro-*m*-5-xylylidine.**

Cryst. from ligroin. M.p. 56°.

*Acetyl*: m.p. 114°.

Ibbotson, Kenner, *J. Chem. Soc.*, 1923, **123**, 1266.

Noelting, Forel, *Ber.*, 1885, **18**, 2679.

3-Nitro-*p*-2-xylydine $C_8H_{10}O_2N_2$ 

MW, 166

Cryst. Volatile in steam.

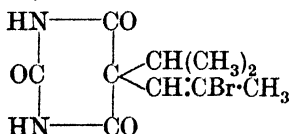
*Benzoyl*: needles from EtOH. M.p. 178°.Hübner, *Ann.*, 1881, **208**, 323.Noelting, Thesmar, Holzach, *Ber.*, 1902, **35**, 640.5-Nitro-*p*-2-xylydine.Plates from EtOH. M.p. 144–5°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. ligroin.*Acetyl*: needles from H<sub>2</sub>O. M.p. 168–9°.*Benzenesulphonyl*: yellow prisms from EtOH.Aq. M.p. 160–3°.*p-Toluenesulphonyl*: pale yellowish cryst. M.p. 185°.Noelting, Witt, Forel, *Ber.*, 1885, **18**, 2667. A.G.F.A., D.R.P., 157,859, (*Chem. Zentr.*, 1905, I, 415).Fisher, Walling, *J. Am. Chem. Soc.*, 1935, **57**, 1701.6-Nitro-*p*-2-xylydine.

Golden-yellow needles from EtOH. M.p. 98°. Sol. boiling EtOH. Sublimes with slight decomp.

*Acetyl*: needles from H<sub>2</sub>O. M.p. 180°.*p-Methylbenzylidene*: pale yellow prisms from EtOH. M.p. 110°.Fittig, Ahrens, Mattheides, *Ann.*, 1868, **147**, 22.Blanksma, *Rec. trav. chim.*, 1905, **24**, 49. v. Kostanecki, *Ber.*, 1886, **19**, 2320.

## Nitroxyllic Acid.

See Nitrodimethylbenzoic Acid.

**Noctal** (*Nostal*, isopropyl-β-bromopropenyl-barbituric acid) $C_{10}H_{13}O_3N_2Br$ 

MW, 289

Colourless cryst. M.p. 178°. Sol. EtOH, Me<sub>2</sub>CO, AcOH. Mod. sol. Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. One of most effective narcotics of barbituric series.Boedecker, Ludwig, *Chem. Abstracts*, 1925, **19**, 1311.

## Nonacosane

 $C_{29}H_{60}$ 

MW, 408

Cryst. from pet. ether. M.p. 63·6–64·1° (62·5–63°). B.p. 346–8°/40 mm., 286°/15 mm., 179°/0 mm.  $n_D^{20}$  1·4361,  $n_D^{25}$  1·43061.Krafft, *Ber.*, 1907, **40**, 4783.Mabery, *Am. Chem. J.*, 1905, **33**, 289.Gluud, *Ber.*, 1919, **52**, 1040.Trost, *Chem. Zentr.*, 1936, I, 1241.

## Nonadecane

 $C_{19}H_{40}$ 

MW, 268

M.p. 32°. B.p. 330°, 248°/100 mm., 226·5°/50 mm., 212°/30 mm., 193°/15 mm. D<sub>4</sub><sup>20</sup> 0·780.Clemmensen, *Ber.*, 1913, **46**, 1841.Schaal, *Ber.*, 1907, **40**, 4787.Hildebrandt, Wachter, *J. Am. Chem. Soc.*, 1929, **51**, 2487.

## Nonadecane-1-carboxylic Acid.

See *n*-Eicosanic Acid.

## Nonadecane-1 : 1-dicarboxylic Acid.

See Octadecylmalonic Acid.

## Nonadecane-1 : 19-dicarboxylic Acid.

See Japanic Acid.

## Nonadecanoic Acid.

See Nonadecylic Acid.

## Nonadecanol-1.

See Nonadecyl Alcohol.

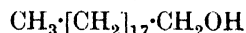
## Nonadecanone-2.

See Methyl heptadecyl Ketone.

## Nonadecanone-4.

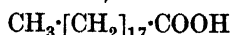
See Propyl pentadecyl Ketone.

## Nonadecanone-10.

Caprinone, *q.v.*Nonadecyl Alcohol (*Nonadecanol*-1) $C_{19}H_{40}O$ 

MW, 284

Cryst. M.p. 62–3° (61·8–62°). B.p. 166–7°/0·32 mm.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.Shiina, *Chem. Zentr.*, 1936, I, 755.Nonadecylic Acid (*Octadecane*-1-carboxylic acid, *nonadecanoic acid*) $C_{19}H_{38}O_2$ 

MW, 298

Leaflets from EtOH. M.p. 66·5°. B.p. 297–8°/100 mm.

Schweizer, *Arch. Pharm.*, 1884, **222**, 770. Oskerkko, *Chem. Zentr.*, 1914, II, 1264.

**Nonadecyl iodide**

$\text{CH}_3\cdot[\text{CH}_2]_{17}\cdot\text{CH}_2\text{I}$   
 $\text{C}_{19}\text{H}_{39}\text{I}$  MW, 394

Cryst. M.p. 42.5–43.5°. B.p. 174–174.5°/  
 0.22 mm.

Shiina, *Chem. Zentr.*, 1936, I, 755.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**,  
 905.

**Nonaldehyde.**

See Pelargonic Aldehyde.

**Nonamethylene.**

See Cyclononane.

**Nonanal.**

See Pelargonic Aldehyde.

**Nonandione-3 : 4.**

See Propionylcaproyl.

**Nonane (1-Ethylheptane)**

$\text{CH}_3\cdot[\text{CH}_2]_7\cdot\text{CH}_3$   
 $\text{C}_9\text{H}_{20}$  MW, 128

Liq. F.p. — 51°. B.p. 149.5°, 86°/100 mm.,  
 59°/30 mm., 39.5°/11 mm.  $D_4^{20}$  0.7177,  $D_{15}^{15}$   
 0.7219.  $n_D^{25}$  1.4025,  $n_D^{20.5}$  1.4165.

Krafft, *Ber.*, 1882, **15**, 1692.

Haller, Lassieur, *Compt. rend.*, 1910, **150**,  
 1017.

Fischer, Klemm, *Chem. Abstracts*, 1930,  
**24**, 4200.

**Nonane-1-carboxylic Acid.**

See *n*-Capric Acid.

**Nonane-2-carboxylic Acid.**

1-Methylpelargonic Acid, *q.v.*

**Nonane-1 : 9-dicarboxylic Acid (Undecane-dioic acid)**

$\text{HOOC}\cdot[\text{CH}_2]_9\cdot\text{COOH}$   
 $\text{C}_{11}\text{H}_{20}\text{O}_4$  MW, 216

Leaflets from  $\text{H}_2\text{O}$ . M.p. 124° (110°). Sol.  
 EtOH, Et<sub>2</sub>O. Spar. sol. hot  $\text{H}_2\text{O}$ .

Dichloride:  $\text{C}_{11}\text{H}_{18}\text{O}_2\text{Cl}_2$ . MW, 253. B.p.  
 191–2°/22 mm.

Diamide:  $\text{C}_{11}\text{H}_{22}\text{O}_2\text{N}_2$ . MW, 214. M.p. 173°.

Dinitrile:  $\text{C}_{11}\text{H}_{18}\text{N}_2$ . MW, 178. Liq. B.p.  
 195°/12 mm.

Monoanilide:  $\text{C}_{17}\text{H}_{25}\text{O}_3\text{N}$ . MW, 291. Cryst.  
 from 50% EtOH. M.p. 112.5–113°.

Dianilide:  $\text{C}_{23}\text{H}_{30}\text{O}_2\text{N}_2$ . MW, 366. Cryst.  
 from EtOH. M.p. 160–1° (156°).

Di-*p*-bromoanilide: m.p. 215°.

Di-*o*-toluidide:  $\text{C}_{25}\text{H}_{34}\text{O}_2\text{N}_2$ . MW, 394. M.p.  
 164°.

Di-*p*-toluidide: m.p. 191°.

$\alpha$ -Anhydride: cryst. from  $\text{C}_6\text{H}_6$ -pet. ether.  
 M.p. 69–70°.

$\beta$ -Anhydride: wax. M.p. 85–8°.

v. Braun, Danziger, *Ber.*, 1912, **45**, 1975.  
 Shukow, Schestakow, *J. prakt. Chem.*,  
 1903, **67**, 416.

Easson, Pyman, *J. Chem. Soc.*, 1931,  
 3000.

Barnicoat, *J. Chem. Soc.*, 1927, 2928.

Hill, Carothers, *J. Am. Chem. Soc.*, 1933,  
**55**, 5027.

**Nonanol-1.**

See Nonyl Alcohol.

**Nonanol-2.**

See Methylheptylcarbinol.

**Nonanol-3.**

See Ethyl-*n*-hexylcarbinol.

**Nonanol-4.**

See Propyl-*n*-amylcarbinol.

**Nonanol-5.**

See Di-*n*-butylcarbinol.

**Nonanol-1-carboxylic Acid.**

Hydroxycapric Acid, *q.v.*

**Nonanol-2-carboxylic Acid.**

See 1-*n*-Heptylhydraacrylic Acid.

**Nonanone-2.**

See Methyl heptyl Ketone.

**Nonanone-3.**

See Ethyl *n*-hexyl Ketone.

**Nonanone-4.**

See Propyl *n*-amyl Ketone.

**Nonanone-5.**

See Di-*n*-butyl Ketone.

**1-Nonene (1-Nonylene, *n*-heptylethylene)**

$\text{CH}_3\cdot[\text{CH}_2]_6\cdot\text{CH}\cdot\text{CH}_2$   
 $\text{C}_9\text{H}_{18}$  MW, 126

B.p. 146°.  $D^{21}$  0.730.  $n_D^{21}$  1.414.

Bourguel, *Bull. soc. chim.*, 1927, **41**, 1475.

Maman, *Chem. Zentr.*, 1936, I, 2332.

**2-Nonene (2-Nonylene, 1-methyl-2-hexyl-ethylene)**

$\text{CH}_3\cdot[\text{CH}_2]_5\cdot\text{CH}\cdot\text{CH}\cdot\text{CH}_3$   
 $\text{C}_9\text{H}_{18}$  MW, 126

Liq. B.p. 149.4–149.9° (147–8°, 148.5°).  $D_{15}^{15}$   
 0.7540,  $D^{21}$  0.738.  $n_D^{21}$  1.420. Odour resembles  
 that of petroleum ether.

Clarke, Adams, *J. Am. Chem. Soc.*, 1915,  
**37**, 2538.

Thoms, Mannich, *Ber.*, 1903, **36**, 2550.

Bourguel, *Bull. soc. chim.*, 1927, **41**,  
 1475.

**4-Nonene** (4-Nonylene, 1-propyl-2-butyl-ethylene)



$\text{C}_9\text{H}_{18}$  MW, 126  
B.p. 144–6°, 44–6°/12 mm.  $D^{18}_D$  0.732.  $n^{18}_D$  1.4212.

Kirrmann, *Compt. rend.*, 1926, **182**, 1629.

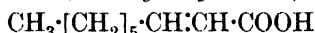
**1-Nonene-1-carboxylic Acid.**

See 1-Decylenic Acid.

**2-Nonene-9-carboxylic Acid.**

See Isodecylenic Acid.

**1-Nonenic Acid** (1-Nonylenic acid, 1-octene-1-carboxylic acid, 2-hexylacrylic acid)



$\text{C}_9\text{H}_{16}\text{O}_2$  MW, 156  
Liq. B.p. 173°/20 mm. Spar. sol.  $\text{H}_2\text{O}$ . Volatile in steam.

Fittig, Schneegans, *Ann.*, 1885, **227**, 80.

Knövenagel, D.R.P., 156,560, (*Chem. Zentr.*, 1905, I, 56).

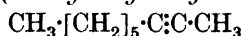
**2-Nonenylethylene.**

See Heptoprene.

**1-Nonine.**

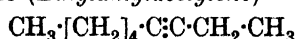
See n-Heptylacetylene.

**2-Nonine** (Methylhexylacetylene)



$\text{C}_9\text{H}_{18}$  MW, 124  
Liq. B.p. 158–9°.  $D^{18}_D$  0.770.  $n^{18}_D$  1.433.  
Truchet, *Ann. chim.*, 1931, **16**, 358.  
Bourguet, *Ann. chim.*, 1925, **3**, 395.

**3-Nonine** (Ethylamylacetylene)



$\text{C}_9\text{H}_{18}$  MW, 124  
B.p. 155–7°.  $D^{20}_D$  0.763.  $n^{20}_D$  1.429.  
Truchet, *Compt. rend.*, 1930, **191**, 854.

**Nonoic Acid.**

See Pelargonic Acid.

**Nonylacetylene** (1-Undecine, rutylicidene)



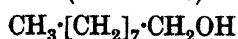
$\text{C}_{11}\text{H}_{20}$  MW, 152  
F.p. – 33°. B.p. 209–10°, 202–4°/745 mm., 110–11°/17 mm., 91°/8 mm.  $D^{25}_D$  0.8666. Sol. most org. solvents. Insol.  $\text{H}_2\text{O}$ .

Thoms, Mannich, *Ber.*, 1903, **36**, 2549.

Bruylants, *Ber.*, 1875, **8**, 413.

Vaughn, *J. Am. Chem. Soc.*, 1933, **55**, 3454.

**Nonyl Alcohol** (Nonanol-1)



$\text{C}_9\text{H}_{20}\text{O}$  MW, 144

Found in orange oil. Yellow liq. F.p. – 5°. B.p. 215°, 107.5°/15 mm., 98–101°/12 mm.  $D^{20}_D$  0.8415,  $D^{20}_D$  0.8279.  $n^{25}_D$  1.43582. Used in rose perfume and cologne waters. Ox. → pelargonic acid.

3 : 5-Dinitrobenzoyl : m.p. 52.2°.

o-Nitrophenylurethane : cryst. from pet. ether. M.p. 34°.

m-Nitrophenylurethane : cryst. from pet. ether. M.p. 66°.

p-Nitrophenylurethane : leaflets from 50% EtOH. M.p. 104°. Sol. EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ ,  $\text{CCl}_4$ , pet. ether.

3 : 5-Dinitrophenylurethane : leaflets from pet. ether. M.p. 60°.

1-Naphthylurethane : m.p. 65.5°.

4'-Iodo-4-diphenylurethane : m.p. 148.4–149.2°.

Krafft, *Ber.*, 1886, **19**, 2221.

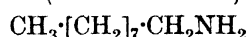
Bouveault, Blanc, D.R.P., 164,294, (*Chem. Zentr.*, 1905, II, 1700).

Harding, Weizmann, *J. Chem. Soc.*, 1910, **97**, 304.

Hoppenbrouwers, *Rec. trav. chim.*, 1932, **51**, 951.

Hoeke, *Rec. trav. chim.*, 1935, **54**, 505.

**Nonylamine** (1-Aminononane)



$\text{C}_9\text{H}_{21}\text{N}$  MW, 143  
B.p. 201°. N-Acetyl : leaflets. M.p. 34–5°. N-Benzoyl : m.p. 49°. Picrate : m.p. 111°.

Thoms, *Chem. Zentr.*, 1901, I, 524.

v. Braun, Sobiecki, *Ber.*, 1911, **44**, 1469.

Mailhe, *Bull. soc. chim.*, 1918, **23**, 235.

Jegorow, *J. prakt. Chem.*, 1912, **86**, 529.

**Nonylene.**

See Nonene.

**Nonylenic Acid.**

See Nonenic Acid.

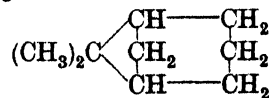
**Nonylic Acid.**

See Pelargonic Acid.

**Nonylic Aldehyde.**

See Pelargonic Aldehyde.

**Nopinane**

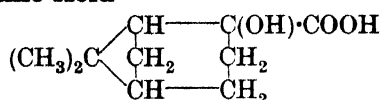


$\text{C}_9\text{H}_{18}$  MW, 124  
B.p. 149°/747 mm.  $D^{22}_D$  0.8611.  $n_D$  1.4641.  
Semmler, Feldstein, *Ber.*, 1914, **47**, 386.

**Nopinene.**

See β-Pinene.

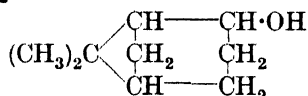
## Nopinic Acid

 $\text{C}_{10}\text{H}_{16}\text{O}_3$ 

MW, 184

*l*-.  
Needles from  $\text{H}_2\text{O}$ . M.p.  $126-8^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{AcOEt}$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin.  $[\alpha]_D^{18} - 16.02^\circ$  in  $\text{Et}_2\text{O}$ .  $\text{PbO}_2$  or  $\text{KMnO}_4$  in  $\text{H}_2\text{SO}_4 \rightarrow$  nopinone.Wallach, Blumann, *Ann.*, 1907, 356, 228.Baeyer, Villiger, *Ber.*, 1896, 29, 1923.Brus, *Compt. rend.*, 1924, 179, 501.

## Nopinol

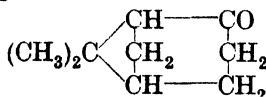
 $\text{C}_9\text{H}_{16}\text{O}$ 

MW, 140

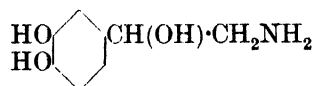
Exists in two forms.

 $\alpha$ -.  
Needles from  $\text{MeOH}$ . M.p.  $101-2^\circ$ . B.p.  $204-5^\circ$ .  $[\alpha]_D^{20} - 9.17^\circ$  in  $\text{EtOH}$ ,  $[\alpha]_D^{18} - 5.32^\circ$  in  $\text{Et}_2\text{O}$ .*Phenylurethane*: m.p.  $131-2^\circ$ . $\beta$ -.  
Viscous liq.  $[\alpha]_D^{17} - 15.03^\circ$  in  $\text{Et}_2\text{O}$ .*Phenylurethane*: m.p.  $95-6^\circ$ .Wallach, Blumann, *Ann.*, 1907, 356, 236.

## Nopinone

 $\text{C}_9\text{H}_{14}\text{O}$ 

MW, 138

*d*-.  
Oil. F.p. about  $0^\circ$ . B.p.  $209^\circ$ ,  $118.2^\circ/43$  mm.,  $87-8^\circ/14$  mm.,  $D_4^{20} 0.9958$ ,  $D_4^{19.6} 0.9807$ ,  $n_D^{20} 1.4787$ .  $[\alpha]_D^{20} + 14.48^\circ$ ,  $+ 38.34^\circ$  in  $\text{MeOH}$ ,  $+ 33.99^\circ$  in  $\text{CHCl}_3$ ,  $+ 11.51^\circ$  in  $\text{Et}_2\text{O}$ ,  $+ 11.35^\circ$  in  $\text{C}_6\text{H}_6$ ,  $+ 8.73^\circ$  in  $\text{CS}_2$ .*Semicarbazone*: needles from  $\text{MeOH}$ . M.p.  $188^\circ$  ( $167^\circ$ ).*Hydrazone*: cryst. from  $\text{AcOEt}$ . M.p.  $42-3^\circ$ .Wallach, Blumann, *Ann.*, 1907, 356, 231.Baeyer, Villiger, *Ber.*, 1896, 29, 1927.Brus, Peyresblauques, *Compt. rend.*, 1929, 187, 984.Schmidt, *Z. angew. Chem.*, 1929, 42, 126.Noradrenalin  
2-aminoethanol,  
catechol(1-[3 : 4-Dihydroxyphenyl]-  
4-[ $\alpha$ -hydroxy- $\beta$ -aminoethyl]- $\text{C}_8\text{H}_{11}\text{O}_3\text{N}$ 

MW, 169

*dl*-.  
Cryst. M.p.  $191^\circ$  decomp. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Sol. dil. acids and alkalis.*B.HCl*: m.p.  $141^\circ$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ . Aq. sol. +  $\text{FeCl}_3$  gives green col.*Oxalate*: m.p.  $175^\circ$ . Sol.  $\text{H}_2\text{O}$ .M.L.B., D.R.P., 157,300, (*Chem. Zentr.*, 1905, I, 315); D.R.P., 193,634, (*Chem. Zentr.*, 1908, I, 430); D.R.P., 195,814, (*Chem. Zentr.*, 1908, I, 1225).

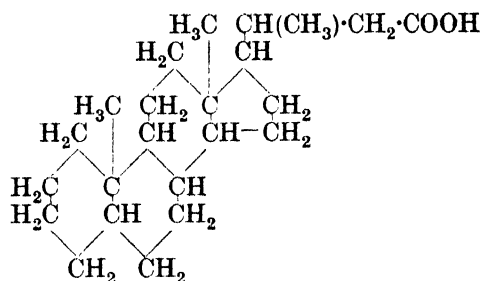
## Noragathic Acid

 $\text{C}_{19}\text{H}_{30}\text{O}_2$ 

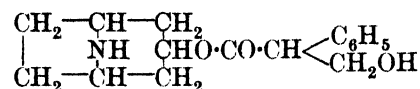
MW, 290

Prisms from  $\text{MeOH}$ . M.p.  $146-7^\circ$ . B.p.  $195-7^\circ/0.9$  mm.,  $180-3^\circ/0.4$  mm.  $[\alpha]_D + 59.3^\circ$  in  $\text{EtOH}$ .*Me ester*:  $\text{C}_{20}\text{H}_{32}\text{O}_2$ . MW, 304. B.p.  $151-2^\circ/0.6$  mm.,  $146-8^\circ/0.1$  mm.  $D_4^{23} 1.002$ .  $n_D^{23} 1.5087$ .  $[\alpha]_D + 57.02^\circ$  in  $\text{EtOH}$ .Ruzicka, Hosking, *Ann.*, 1929, 469, 188; *Helv. Chim. Acta.*, 1931, 14, 214.

## Norallocholanolic Acid

 $\text{C}_{23}\text{H}_{38}\text{O}_2$ 

MW, 346

Plates from  $\text{Me}_2\text{CO}$ . M.p.  $170-170.5^\circ$ .*Me ester*:  $\text{C}_{24}\text{H}_{40}\text{O}_2$ . MW, 360. M.p.  $78-9^\circ$ .Chuang, *Ann.*, 1933, 500, 280.Heilbron, Samant, Simpson, *J. Chem. Soc.*, 1933, 1413.Noratropine (Nortropine ester of tropic acid,  
*dl*-norhyoscyamine) $\text{C}_{16}\text{H}_{21}\text{O}_3\text{N}$ 

MW, 275



Cryst. from  $\text{Me}_2\text{CO}$ . M.p. 113–14°. Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ ,  $\text{AcOEt}$ . Mod. sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{H}_2\text{O}$ . Optically inactive.  $\text{CH}_3\text{I} \rightarrow$  atropine.

$B, H_2O$ : m.p. 73°.

$B, HCl$ : m.p. 193°. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Prac. insol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ .

$B_2, H_2SO_4$ : needles from  $\text{H}_2\text{O}$ . M.p. 257°. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Prac. insol.  $\text{Me}_2\text{CO}$ .

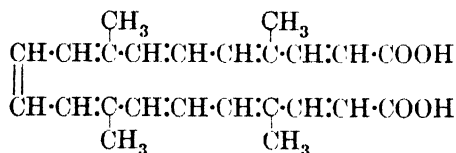
$B_2, (COOH)_2$ : cryst. from  $\text{H}_2\text{O}$ . M.p. 247–8°.

$B, HAuCl_4$ : yellow needles from dil.  $\text{EtOH}$ . M.p. 157°. Sol.  $\text{EtOH}$ . Spar. sol.  $\text{H}_2\text{O}$ .

*Picrate*: needles. M.p. 227°.

Carr, Reynolds, *J. Chem. Soc.*, 1912, **101**, 955.

### Norbixin



$\text{C}_{24}\text{H}_{28}\text{O}_4$

MW, 380

*Cis*:

Needles from  $\text{AcOH}$ . M.p. 254–5°. Sol.  $\text{MeOH}$ ,  $\text{AcOH}$ , hot  $\text{EtOH}$ , aq. alkalis. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{AcOEt}$ .

*Me ester*: see Bixin.

*Di-Me ester*: see under Bixin.

*Trans*:

See Isonorbixin.

Kuhn, Grundmann, *Ber.*, 1932, **65**, 1880.

Kuhn, Winterstein, *ibid.*, 646.

Herzig, Faltis, *Ann.*, 1923, **431**, 40.

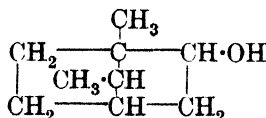
Vieböck, *Ber.*, 1934, **67**, 377.

Karrer, Helfenstein, Widmer, van Itallie, *Helv. Chim. Acta*, 1929, **12**, 743, 752.

### Norborneol

The following compounds have been described under this name:

(a)



$\text{C}_9\text{H}_{16}\text{O}$

MW, 140

(i) *Santenone alcohol*.

Isolated from East Indian sandalwood oil. M.p. 58–62°.  $\text{CrO}_3 \rightarrow$  santenone.

Schimmel, *Bericht vom Oktober*, 1910, 100; *Chem. Zentr.*, 1910, II, 1757.

(ii) *Santenol* (N.B. The nomenclature of the santenols is confused).

M.p. 68–70°. B.p. 195–6°, 87–8°/9 mm.  $\text{CrO}_3 \rightarrow$  santenone.  $\text{PCl}_5 \rightarrow$  norbornyl chloride.  $\text{KMnO}_4 \rightarrow$  santenic acid.

*Formate*: b.p. 87–94°/9 mm.  $D^{20}_D$  1.0092.  $n_D$  1.46559.

*Acetate*: b.p. 89–90.5°/9 mm.  $D^{20}_D$  0.987.  $n_D$  1.45962.

Semmler, Bartelt, *Ber.*, 1907, **40**, 4467; 1908, **41**, 128, 389.

Aschan, *Ber.*, 1907, **40**, 4923.

(iii) *Norisoborneol*,  $\alpha$ -santenol (?).

M.p. 97–8° (91–2°). B.p. 88°/9 mm.  $\text{CrO}_3 \rightarrow$  santenone.

*Phenylurethane*: m.p. 61–2°.

Semmler, Bartelt, *Ber.*, 1907, **40**, 4469.

Aschan, *Ber.*, 1907, **40**, 4923.

Deussen, *J. prakt. Chem.*, 1926, **114**, 114.

(iv)  $\alpha$ -Santenol.

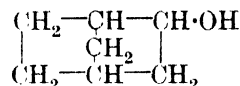
Needles. M.p. 86°.

*Hydrogen diphenate*: cryst. from dil.  $\text{EtOH}$ . M.p. 119–20°.

Komppa, *Ber.*, 1929, **62**, 1751.

Diels, Alder, Petersen, *Ann.*, 1931, **486**, 210.

(b)



$\text{C}_7\text{H}_{12}\text{O}$

MW, 112

(v)  $\alpha$ -Norborneol, endonorborneol.

M.p. 149–50°.

*Phenylurethane*: m.p. 158–9°.

Komppa, Beckmann, *Ann.*, 1934, **512**, 177.

(vi)  $\beta$ -Norborneol, exonorborneol.

M.p. 127–8° (123–4°). B.p. 176–7°.  $\text{K}_2\text{Cr}_2\text{O}_7 \rightarrow$  norcamphor.

*Phenylurethane*: m.p. 147° (144–5°).

*Hydrogen phthalate*: m.p. 102–3°.

*Acetate*: b.p. 89–90°/20 mm.

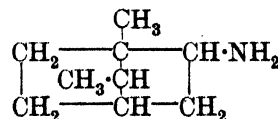
Komppa, Beckmann, *Ann.*, 1934, **512**, 181.

Alder, Stein, Rolland, Schulze, *Ann.*, 1934, **514**, 220, 226.

### Norbornylamine

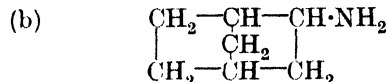
The following compounds have been described under this name:

(a)



$\text{C}_9\text{H}_{17}\text{N}$

MW, 139

(i) *Santenylamine*.B.p. 69°/10 mm.  $D^{20}_D$  0.9163.  $n^{20}_D$  1.47642.*B.HCl*: m.p. 272° decomp.*Picrate*: m.p. 208°.*Oxalate*: decomp. at 280°.Semmler, Bartelt, *Ber.*, 1908, **41**, 127. $C_7H_{13}N$ 

MW, 111

(ii)  $\beta$ -Norbornylamine.

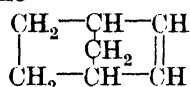
M.p. 75–80°. B.p. 156–7°.

*B.HCl*: m.p. about 260° decomp.*Picrate*: m.p. 174–5°.*B.HAuCl<sub>4</sub>*: m.p. 211–12° decomp.*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: decomp. above 200°.*Carbamide*: m.p. 196–7°.*Phenylthiocarbamide*: m.p. 154–5°.Komppa, Beckmann, *Ann.*, 1934, **512**, 180.(iii) *Endonorbornylamine*.*B.HCl*: m.p. 295°.*Acetyl*: m.p. 124°.Alder, Stein, Rolland, Schulze, *Ann.*, 1934, **514**, 215, 224.(iv) *Exonorbornylamine*.*B.HCl*: does not melt below 345°.*Acetyl*: m.p. 139°.

See previous reference.

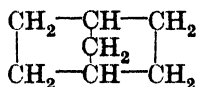
**Norbornylane.**

See Norcamphane.

**Norbornylene** $C_7H_{10}$ 

MW, 94

M.p. 52–4°. Sublimes.

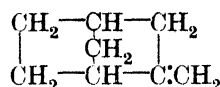
*Dibromide*: liq.*Nitroschloride*: needles from  $\text{CHCl}_3$ . M.p. 157–8°. $C_6H_8N_3$  comp.: m.p. 101–2°.Komppa, Beckmann, *Ann.*, 1934, **512**, 184.**Norcamphane** (*Norbornylane*, 1 : 2 : 2-bi-cycloheptane) $C_7H_{12}$ 

MW, 96

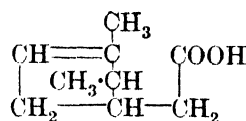
M.p. 86–7°. Sublimes.

Komppa, Beckmann, *Ann.*, 1934, **512**, 183.

Dict. of Org. Comp.—III.

**Norcamphene** $C_8H_{12}$ 

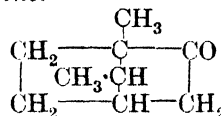
MW, 108

B.p. 123°/755 mm.  $D^{13}_D$  0.8789.*Nitroschloride*: cryst. from AcOEt. M.p. 125°.Diels, Alder *et al.*, *Ann.*, 1929, **470**, 79.**Norcampholenic Acid** $C_9H_{14}O_2$ 

MW, 154

B.p. 132–4°/10 mm.  $D^{20}_D$  1.014.  $n_D$  1.47936.*Nitrile*:  $C_9H_{13}N$ . MW, 135. B.p. 82–3°/9 mm.  $D^{20}_D$  0.950.  $n_D$  1.4720.Semmler, Bartelt, *Ber.*, 1908, **41**, 127.**Norcamphor**

Two compounds have been described under this name:

(i) *Santenone*. $C_9H_{14}O$ 

MW, 138

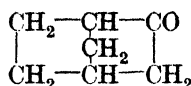
l.

Occurs in East Indian sandalwood oil. M.p. 58–61°. B.p. 193–5°.  $[\alpha]_D - 4.40^\circ$  in EtOH.*Semicarbazone*: m.p. 222–4°.*Oxime*: b.p. 110–13°/6 mm.

dl.

M.p. 55–7° (50–2°, 30°). B.p. 196.5–197°, 75–6°/9 mm.  $D^{20}_D$  0.966.  $n^{20}_D$  1.4690.  $\text{KMnO}_4 \rightarrow$  santenic acid.  $\text{Na} + \text{EtOH} \rightarrow$  norisoborneol.*Semicarbazone*: m.p. 228–9° (224°).*Oxime*: cryst. from pet. ether. M.p. 80–1°. B.p. 116–20°/9 mm.*Oxymethylene deriv.*: b.p. 110–13°/9 mm.  $D^{20}_D$  1.066.  $n_D$  1.50045.*Benzylidene deriv.*: b.p. 182–4°/10 mm.  $D^{20}_D$  1.041.  $n_D$  1.57516.Semmler, Bartelt, *Ber.*, 1907, **40**, 4467; 1908, **41**, 127.Schimmel, *Chem. Zentr.*, 1910, II, 1757.Rimini, *Gazz. chim. ital.*, 1913, **43**, 522.

(ii)

 $\text{C}_7\text{H}_{10}\text{O}$ 

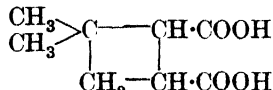
MW, 110

Cryst. M.p. 93–4° (91–2°). Mod. sol.  $\text{H}_2\text{O}$ .

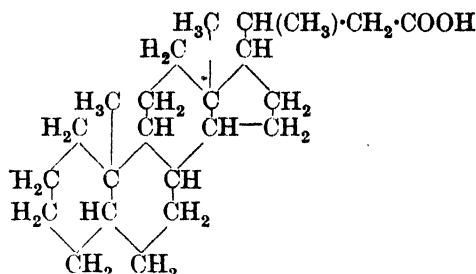
Semicarbazone: m.p. 196.5–197.5°. Sol.

EtOH. Spar. sol.  $\text{C}_6\text{H}_6$ .Hintikka, Komppa, *Chem. Zentr.*, 1918, II, 370.Komppa, *Ber.*, 1909, 42, 898 (Footnote).Diels, Alder *et al.*, *Ann.*, 1929, 470, 76.**Norcamphoric Acid.**

See Apocamphoric Acid.

**Norcaryophyllenic Acid** (3 : 3-Dimethylcyclobutane-1 : 2-dicarboxylic acid) $\text{C}_8\text{H}_{12}\text{O}_4$ 

MW, 172

*Cis form* :*d.*Cryst. from  $\text{C}_6\text{H}_6$  or dil. HCl. Sinters at 118°. M.p. 125–7° (123.5–124.5°). Sol.  $\text{H}_2\text{O}$  and usual org. solvents.  $[\alpha]_{5461}^{20} + 137^\circ$  in  $\text{CHCl}_3$ ,  $[\alpha]_{\text{D}}^{20} + 118^\circ$  in  $\text{CHCl}_3$ .*Di-Me ester*:  $\text{C}_{10}\text{H}_{16}\text{O}_4$ . MW, 200. B.p. 107°/12 mm.*Anhydride*:  $\text{C}_{16}\text{H}_{22}\text{O}_7$ . MW, 326. M.p. 38–9°.*Chloride*:  $\text{C}_8\text{H}_{11}\text{O}_3\text{Cl}$ . MW, 190.5. B.p. 99–100°/12 mm.*dl.*Cryst. from  $\text{H}_2\text{O}$ . M.p. 148–9°.*Anhydride*: m.p. 40–1°. B.p. 100–2°/1 mm.Evans, Ramage, Simonsen, *J. Chem. Soc.*, 1934, 1809.Ramage, Simonsen, *J. Chem. Soc.*, 1935, 532.Ruzicka, Zimmermann, *Helv. Chim. Acta*, 1935, 18, 219.Rydon, *J. Chem. Soc.*, 1936, 593.**Norcephæline** (Noremeline, emetoline) $\text{C}_{25}\text{H}_{32}\text{O}_4\text{N}_2$  MW, 424M.p. about 205°. Sol. EtOH, acids and alkalis. Spar. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ .*B,2HCl*: amorph. powder. M.p. about 240° decomp. Sol. MeOH. Mod. sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{Me}_2\text{CO}$ .Karrer, *Ber.*, 1916, 49, 2057.Carr, Pyman, *J. Chem. Soc.*, 1914, 105, 1616.Pyman, *J. Chem. Soc.*, 1917, 111, 1127.Hesse, *Ann.*, 1914, 405, 27.**Norcholanic Acid** $\text{C}_{23}\text{H}_{38}\text{O}_2$ 

MW, 346

Needles from AcOH. M.p. 177°.

*Me ester*:  $\text{C}_{24}\text{H}_{40}\text{O}_2$ . MW, 360. Needles from MeOH. M.p. 74°.*Et ester*:  $\text{C}_{25}\text{H}_{42}\text{O}_2$ . MW, 374. Prisms from EtOH. M.p. 66–7°.Wieland, Schlichting, Jacobi, *Z. physiol. Chem.*, 1926, 161, 94.**Norcitronellol.**

See 6-Methyl-2-octenol-8.

**Norcodeine** (Normorphine methyl ether) $\text{C}_{17}\text{H}_{19}\text{O}_3\text{N}$ 

MW, 285

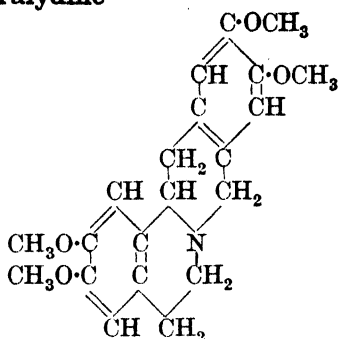
Needles from AcOEt. M.p. 186°.

*B,HCl*: cryst. +  $3\text{H}_2\text{O}$ . M.p. anhyd. 309°.*B,2H,PtCl\_6*: yellow leaflets. M.p. 239°.*B,HI*: m.p. 257°.*N-β-Phenylethyl*:  $\text{C}_{25}\text{H}_{27}\text{O}_3\text{N}$ . MW, 389.Cryst. M.p. 114°. Very sol. EtOH. Mod. sol.  $\text{Et}_2\text{O}$ . Spar. sol. pet. ether. *B,HCl*: cryst. M.p. 277°. *B,2H,PtCl\_6*: m.p. 216–17°.*N-Cyano*: cryst. from EtOH. M.p. 263°. Sol. EtOH. Mod. sol.  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ . *Acetyl*: cryst. from EtOH. M.p. 184°. Sol.  $\text{CHCl}_3$ . Mod. sol.  $\text{Et}_2\text{O}$ . Sol. 20 parts boiling, 200 parts cold EtOH.*N-Nitroso*: prisms from EtOH.Aq. M.p. 246°.*N-Me*: Codeine, *q.v.*Speyer, Walther, *Ber.*, 1930, 63, 854.  
v. Braun, *Ber.*, 1914, 47, 2320.**Norconessine** $\text{C}_{23}\text{H}_{38}\text{N}_2$ 

MW, 342

Occurs in seeds of *Holorrhena antidiysenterica*. Dextrorotatory. B.p. 238–40°/0.7 mm.*Di-acid oxalate*: m.p. 225–7° decomp. Sol.  $\text{H}_2\text{O}$ , EtOH.*B,2HCl*: m.p. 340° decomp. Sol.  $\text{H}_2\text{O}$ , EtOH.*Dimethiodide*: m.p. 310–12° decomp.Haworth, *J. Chem. Soc.*, 1932, 631.

## Norcoralydine

 $C_{21}H_{25}O_4N$ 

MW, 355

Plates from dil. EtOH. M.p. 157–8° (151.5–152.5°). Sol. cold dil.  $H_2SO_4$ , EtOH,  $CHCl_3$ . Spar. sol.  $C_6H_6$ . Turns yellow in air. I  $\rightarrow$  dehydronorcoralydine.  $KMnO_4 \rightarrow$  metahemipinic acid.

$B, HCl$ : needles from  $HCl.Aq.$  M.p. 213°.

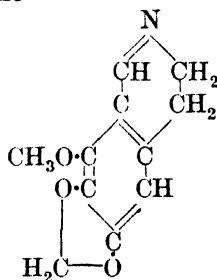
$B_2, H_2PtCl_6$ : orange-red prisms from EtOH. M.p. about 231°.

*Picrate*: yellow needles from EtOH. M.p. 138°. Spar. sol.  $H_2O$ .

Späth, Kruta, *Monatsh.*, 1928, **50**, 341.

Pictet, Chou, *Ber.*, 1916, **49**, 370.

## Norcotarnine

 $C_{11}H_{11}O_3N$ 

MW, 205

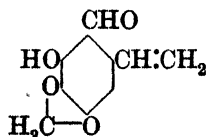
Yellow flocks from ligroin.

*Picrate*: yellow needles from EtOH. M.p. 182–4°.

*Methiodide*: cotarnine hydriodide. M.p. 184–6° decomp.

Decker, Becker, *Ann.*, 1913, **395**, 330.

**Norcotarnone** (6-Hydroxy-4:5-methylene-dioxy-2-vinylbenzaldehyde, 2-hydroxy-6-vinyl-piperonal)

 $C_{10}H_8O_4$ 

MW, 192

Yellowish-green cryst. from EtOH. M.p. 89°.

*K salt*: yellow scales. Spar. sol. cold  $H_2O$ .

*Me ether*: cotarnone.  $C_{11}H_{10}O_4$ . MW, 206. Plates from EtOH. M.p. 78°. Part. volatile in steam. Insol. cold  $H_2O$ . Mod. sol. EtOH,  $Et_2O$ , AcOH.  $KMnO_4 \rightarrow$  cotarnic acid. *Oxime*: needles from dil. EtOH. M.p. 130–2°.

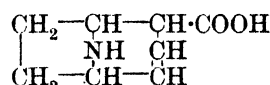
*Acetyl*: needles from EtOH or AcOH. M.p. 84–5°.

*Oxime*: plates from EtOH. M.p. 202–3°. *O-Acetyl*: m.p. 115–16°. *Diacetyl*: m.p. 100–1°.

Freund, Baker, *Ber.*, 1903, **36**, 1530.

Roser, *Ann.*, 1888, **249**, 163.

## Norecgonidine

 $C_8H_{11}O_2N$ 

MW, 153

M.p. 254–5°. Sol.  $H_2O$ , EtOH. Neutral.

$B, HCl$ : m.p. 257°. Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .

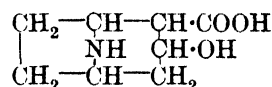
*Et ester*:  $C_{10}H_{15}O_2N$ . MW 181. B.p. 157°/25 mm.  $B, H, AuCl_4$ : m.p. 133°.

$B_2, H_2PtCl_6$ : m.p. 251°.

$B, H, AuCl_4$ : m.p. 204°.

v. Braun, Müller, *Ber.*, 1918, **51**, 247.

## Norecgonine

 $C_8H_{13}O_3N$ 

MW, 171

*l.*

Needles. M.p. 233°. Sol.  $H_2O$ .

$B, H, AuCl_4$ : yellow needles. M.p. 211°. Spar. sol.  $H_2O$ .

*O-Benzoyl*: m.p. 230°.  $B, HCl, 2H_2O$ : m.p. 217–18°.  $B, H, AuCl_4$ : yellow needles. M.p. 228° decomp. *Me ester*: liq.  $B, H, AuCl_4$ : needles from  $H_2O$ . M.p. 181–2°. *Et ester*: liq.  $B, H, AuCl_4$ : m.p. 160–5°.

*N-Me*: see *l*-Ecgonine.

Einhorn, *Ber.*, 1888, **21**, 3029.

See also Willstätter, Müller, *Ber.*, 1898, **31**, 2655.

## Nor-ψ-ecgonine.

*d.*

Needles from EtOH- $Et_2O$ .  $MeI \rightarrow d$ -ψ-ecgonine.

*Me ester*:  $C_9H_{15}O_3N$ . MW, 185. Cryst. from  $AcOEt$  or  $C_6H_6$ . M.p. 160°. Sol.  $MeOH$ , EtOH. Insol.  $Et_2O$ , ligroin.

*Et ester*:  $C_{10}H_{17}O_3N$ . MW, 199. Needles.

from AcOEt. M.p. 137°. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

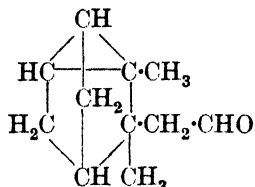
O-Benzoyl: needles from dil. EtOH. Et ester: needles from EtOH. M.p. 127°. Sol. CHCl<sub>3</sub>, AcOEt, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH. Insol. CS<sub>2</sub>, ligroin. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: m.p. 142°.

N-Me: see d-ψ-Ecgonine.

Einhorn, Friedländer, *Ber.*, 1893, **26**, 1482.

See also Willstätter, Müller, *Ber.*, 1898, **31**, 2655.

### Norecsantalal (Nortricycloekasantalal)



C<sub>11</sub>H<sub>16</sub>O

MW, 164

Occurs in East Indian sandalwood oil. B.p. 222-4°, 92-4°/11 mm., 86-7°/6 mm. D<sub>20</sub> 0.9938 (0.9964). n<sub>D</sub><sup>20</sup> 1.48393. Na + EtOH → norecsantalol. NH<sub>3</sub>·AgNO<sub>3</sub> → norecsantalic acid.

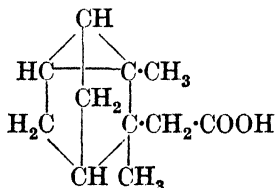
Oxime: b.p. 142-4°/10 mm., 135-7°/7 mm.

Semicarbazone: m.p. 224° (216°). Spar. sol. EtOH.

Semmler, *Ber.*, 1909, **42**, 588; 1910, **43**, 1724, 1893.

Semmler, Zaar, *ibid.*, 1891.

### Norecsantallic Acid (Nortricycloekasantalic acid)



C<sub>11</sub>H<sub>16</sub>O<sub>2</sub>

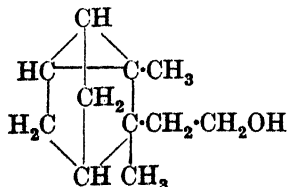
MW, 180

Cryst. from dil. EtOH. M.p. 93°. B.p. 143-5°/10 mm. [α]<sub>D</sub> - 33° 17' in EtOH.

Me ester: C<sub>12</sub>H<sub>18</sub>O<sub>2</sub>. MW, 194. B.p. 102-4°/10 mm. D<sub>20</sub> 1.0228. n<sub>D</sub> 1.47348.

Semmler, *Ber.*, 1910, **43**, 1724, 1893.

### Norecsantalol (Nortricycloekasantalol)



C<sub>11</sub>H<sub>18</sub>O

MW, 166

B.p. 114-17°/10 mm. D<sub>20</sub> 0.9958. n<sub>D</sub> 1.49049. CrO<sub>3</sub> → norecsantalal.

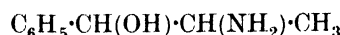
Semmler, Zaar, *Ber.*, 1910, **43**, 1890.

Semmler, *ibid.*, 1893.

### Noremetine.

See Norcephaeline.

**Norephedrine** (2-Amino-1-phenylpropanol-1, α-hydroxy-β-aminopropylbenzene)



C<sub>9</sub>H<sub>13</sub>ON

MW, 151

l.

Occurs in Ma Huang and European *Ephedra*. M.p. about 50°. [α]<sub>D</sub><sup>20</sup> - 14.56° in EtOH.

B.HCl: m.p. 171-2°. [α]<sub>D</sub><sup>20</sup> - 33.27° in H<sub>2</sub>O.

B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>·2H<sub>2</sub>O: m.p. anhyd. 285-6° decomp. [α]<sub>D</sub><sup>20</sup> - 31.99° in H<sub>2</sub>O.

Oxalate: m.p. 245° decomp.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: m.p. 221° decomp.

B.HAuCl<sub>4</sub>: m.p. 188°.

N-p-Nitrobenzoyl: m.p. 175-6°. [α]<sub>D</sub><sup>23</sup> - 49.58° in CHCl<sub>3</sub>.

Hydrogen tartrate + EtOH: softens at 130°. M.p. about 160°. [α]<sub>D</sub><sup>20</sup> - 34.46° (EtOH free).

d.

M.p. 52°. [α]<sub>D</sub><sup>27</sup> + 14.76° in EtOH.

B.HCl: m.p. 171-2°. [α]<sub>D</sub><sup>27</sup> + 33.4° in EtOH.

B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>·2H<sub>2</sub>O: m.p. anhyd. 285-6° decomp. [α]<sub>D</sub><sup>27</sup> + 31.51°.

Oxalate: m.p. 245°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: m.p. 221.5° decomp.

B.HAuCl<sub>4</sub>: m.p. 188°.

N-p-Nitrobenzoyl: m.p. 175-6°. [α]<sub>D</sub><sup>20</sup> + 49.95° in CHCl<sub>3</sub>.

Hydrogen tartrate + EtOH: m.p. about 160°. [α]<sub>D</sub><sup>27</sup> + 34.69° (EtOH free).

dl.

Plates from Et<sub>2</sub>O. M.p. 104-5°.

B.HCl: m.p. 194° (192°).

B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>: m.p. 285-6°.

Hydrogen oxalate: m.p. 245° decomp.

Dioxalate: m.p. 182-3° decomp.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: yellow needles from H<sub>2</sub>O. M.p. 221.5° decomp.

N-Acetyl: m.p. 135°.

N-Benzoyl: m.p. 143°.

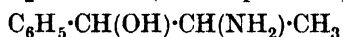
O: N-Dibenzoyl: m.p. 167-8°.

N-p-Nitrobenzoyl: cryst. from EtOH. M.p. 189°.

Kanao, *Ber.*, 1930, **63**, 95.

Nagai, Kanao, *Ann.*, 1929, **470**, 157.

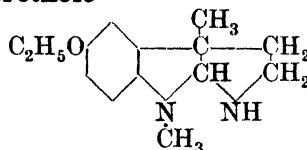
Hey, *J. Chem. Soc.*, 1930, 1232.

**Nor- $\psi$ -ephedrine** (*Norisoephedrine*) $\text{C}_9\text{H}_{13}\text{ON}$ 

MW, 151

*l.*M.p. 77.5–78°.  $[\alpha]_D^{20} - 32.64^\circ$  in EtOH.*B, HCl*: m.p. 180–1°.  $[\alpha]_D^{20} - 42.68^\circ$  in  $\text{H}_2\text{O}$ .*B, H<sub>2</sub>SO<sub>4</sub>*: m.p. 290–1° decomp.  $[\alpha]_D^{20} - 39.99^\circ$ .*Oxalate*: m.p. 235°.*B, H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 199°.*B, H<sub>2</sub>AuCl<sub>4</sub>*: m.p. 137–8°.*N-p-Nitrobenzoyl*: m.p. 199°.  $[\alpha]_D^{20} - 105.13^\circ$  in  $\text{CHCl}_3$ . *B, HCl*: m.p. 246°.  $[\alpha]_D^{22} - 54.63^\circ$  in  $\text{H}_2\text{O}$ .*Hydrogen tartrate*: m.p. 202° decomp.  $[\alpha]_D^{20} - 13.39^\circ$  in  $\text{H}_2\text{O}$ .*dl.*Occurs in Ma Huang. M.p. 77° (77.5–80°).  $[\alpha]_D^{20} + 33.14^\circ$  in EtOH,  $+ 24.7^\circ$  in  $\text{H}_2\text{O}$ ,  $[\alpha]_{5461}^{20} + 32.2^\circ$  in MeOH.*B, HCl*: m.p. 180–1°.  $[\alpha]_D^{20} + 42.53^\circ$  in  $\text{H}_2\text{O}$ . *B, H<sub>2</sub>SO<sub>4</sub>*: m.p. 295° decomp.  $[\alpha]_D^{20} + 40.12^\circ$  in  $\text{H}_2\text{O}$ ,  $[\alpha]_{5461}^{20} + 42.9^\circ$  in  $\text{H}_2\text{O}$ .*Oxalate*: m.p. 235° decomp.*B, H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 198°.*B, H<sub>2</sub>AuCl<sub>4</sub>*: m.p. 137–8°.*N-Benzenesulphonyl*: m.p. 103–4°.*N-Benzoyl*: m.p. 132°.  $[\alpha]_{5461}^{20} + 58.3^\circ$  in MeOH.*O:N-Dibenzoyl*: m.p. 156–7°.  $[\alpha]_{5461}^{20} + 28^\circ$  in MeOH.*N-p-Nitrobenzoyl*: m.p. 199.5°.  $[\alpha]_D^{20} + 104.96^\circ$  in  $\text{CHCl}_3$ .*O-Benzoyl, HCl*: m.p. 244.5° decomp.  $[\alpha]_{5461}^{20} - 32.5^\circ$  in  $\text{H}_2\text{O}$ .*Hydrogen tartrate*: m.p. 202°.  $[\alpha]_D^{20} + 13.36^\circ$  in  $\text{H}_2\text{O}$ .*dl.*

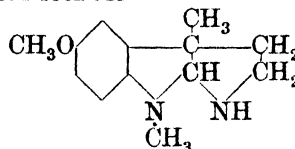
Plates from pet. ether. M.p. 71°.

*B, HCl*: m.p. 169°.*B, H<sub>2</sub>SO<sub>4</sub>*: m.p. 290–1° decomp.*Hydrogen oxalate*: m.p. 235°.*Dioxalate*: m.p. 171° decomp.*B, H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 199.5° decomp.*B, H<sub>2</sub>AuCl<sub>4</sub>*: m.p. 132–3°.*N-Acetyl*: m.p. 85–6°.*N-p-Nitrobenzoyl*: m.p. 170°.*O-Benzoyl, B, HCl*, m.p. 220°. *B, H<sub>2</sub>SO<sub>4</sub>*: m.p. 182° decomp. *Picrate*: m.p. 186° decomp.Smith, *J. Chem. Soc.*, 1928, 51.Kanao, *Ber.*, 1930, 63, 98.Nagai, Kanao, *Ann.*, 1929, 470, 157.Hey, *J. Chem. Soc.*, 1930, 1232.Gibson, Levin, *J. Chem. Soc.*, 1929, 2754.  $\text{C}_{11}\text{H}_8\text{N}_2$ **Noreserethole** $\text{C}_{14}\text{H}_{20}\text{ON}_2$ 

MW, 232

*d.**d-Hydrogen tartrate*: m.p. 188–9°.  $[\alpha]_D^{28} + 202.1^\circ$  in  $\text{H}_2\text{O}$ .*l.**d-Hydrogen tartrate*: m.p. 190–1°.  $[\alpha]_D^{28} - 53.3^\circ$  in  $\text{H}_2\text{O}$ .*dl.*

B.p. 187–92°/12 mm.

*B, HCl*: m.p. 191–2°.*Benzoyl*: m.p. 108°.*B, H<sub>2</sub>PtCl<sub>6</sub>, 1/2 H<sub>2</sub>O*: m.p. 185° decomp.*Picrate*: orange-red prisms from EtOH. M.p. 180–1° (184–5°, 191–2°).*Picrolonate*: m.p. 221° (227°).Robinson, Sugimoto, *J. Chem. Soc.*, 1932, 314.Hoshino, Kobayashi, *Ann.*, 1935, 516, 88.Julian, Pikel, *J. Am. Chem. Soc.*, 1935, 57, 563.**Noresermethole** $\text{C}_{13}\text{H}_{18}\text{ON}_2$ 

MW, 218

*dl.*

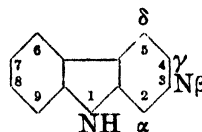
B.p. 130–2° in high vacuum.

*Picrate*: orange-red prisms from EtOH. M.p. 162–3°.*Picrolonate, 1/2 H<sub>2</sub>O*: m.p. 227° decomp.King, Robinson, *J. Chem. Soc.*, 1932, 1433.King, Liguori, Robinson, *J. Chem. Soc.*, 1934, 1416.**Norgranatanine.**

See Granatanine.

**Norgranatoline.**

See footnote under Granatoline.

**Norharman** ( $\beta$ -Carboline, 3-carboline)

MW, 168

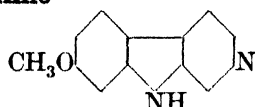
Colourless needles. M.p. 198.5°. Sol. EtOH, Et<sub>2</sub>O, AcOEt, MeOH, hot H<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>, pet. ether. Blue fluor. in dil. acid sol.

*Picrate*: m.p. 260° decomp.

Kermack, Perkin, Robinson, *J. Chem. Soc.*, 1921, 119, 1602.

Gulland, Robinson, Scott, Thornley, *J. Chem. Soc.*, 1929, 2926 (*Footnote*).

### Norharmine



Probable structure

C<sub>12</sub>H<sub>10</sub>ON<sub>2</sub> MW, 198

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 218°. Sol. hot EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Sublimes without decomp. Slowly colours pine shaving + HCl.

*Hydrochloride*: pale yellow prisms from H<sub>2</sub>O. Dil. sols. show intense blue fluor.

*Aurichloride*: pale brown needles from EtOH.

*Mercurichloride*: pale yellow needles from H<sub>2</sub>O.

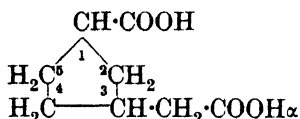
Perkin, Robinson, *J. Chem. Soc.*, 1912, 101, 1785.

Kermack, Perkin, Robinson, *J. Chem. Soc.*, 1921, 119, 1619.

### Norhemipinic Acid.

See 3: 4-Dihydroxyphthalic Acid.

**Norhomocamphoric Acid** (*Cyclopentane-1-carboxylic acid-3-acetic acid, 3-carboxycyclopentyl-acetic acid*)



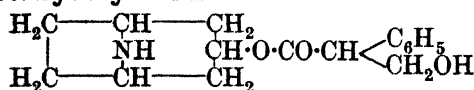
C<sub>8</sub>H<sub>12</sub>O<sub>4</sub> MW, 172

Cryst. from Et<sub>2</sub>O, H<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>. M.p. 137–137.5°. Sol. EtOH, Et<sub>2</sub>O.

1-Et ester α-Me ester: C<sub>11</sub>H<sub>18</sub>O<sub>4</sub>. MW, 214. B.p. 135–41°/13 mm. D<sub>4</sub><sup>17</sup> 1.0683. n<sub>D</sub><sup>17</sup> 1.4505.

Hintikka, Komppa, *Chem. Zentr.*, 1918, II, 370.

### Norhyoscyamine



C<sub>18</sub>H<sub>21</sub>O<sub>3</sub>N MW, 275

*l.*

Found in bark of *Scopolia Japonica*, and *Mandragora Officinarum*. Needles from CHCl<sub>3</sub>-Et<sub>2</sub>O. M.p. 140.5° (133–4°). Sol. EtOH, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O, Me<sub>2</sub>CO. Sol. 270 parts H<sub>2</sub>O. [α]<sub>D</sub><sup>20</sup> – 21.2° in EtOH.

*B, HCl*: needles from EtOH-Et<sub>2</sub>O. M.p. 267°. B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>: needles + 3H<sub>2</sub>O from Me<sub>2</sub>CO.Aq. M.p. 249°. Sol. 5 parts H<sub>2</sub>O at 15°.

*Oxalate*: prisms from Me<sub>2</sub>CO.Aq. M.p. 245–6°. Sol. 20 parts H<sub>2</sub>O at 15°.

*B, H<sub>2</sub>AuCl<sub>4</sub>*: yellow leaflets. M.p. 178–9° (174°). Mod. sol. hot H<sub>2</sub>O.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: needles. M.p. 141°.

*Picrate*: m.p. 220°.

N-Me: see Hyoscyamine.

*dl.*

See Noratropine.

Carr, Reynolds, *J. Chem. Soc.*, 1912, 101, 946.

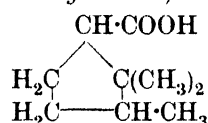
Merck, *Arch. Pharm.*, 1903, 231, 117.

Hesse, *J. prakt. Chem.*, 1901, 64, 276.

### Norisoborneol.

See under Norborneol.

**Norisocampholic Acid** (2: 2: 3-Trimethylcyclopentane-1-carboxylic acid)



C<sub>9</sub>H<sub>16</sub>O<sub>2</sub> MW, 156

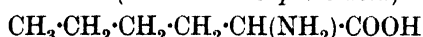
Liq. B.p. 130°/12 mm. D<sub>4</sub><sup>18</sup> 0.9995. n<sub>D</sub><sup>19</sup> 1.4587.

*Chloride*: C<sub>9</sub>H<sub>15</sub>OCl. MW, 174.5. B.p. 85°/13 mm.

*Ethylamide*: C<sub>11</sub>H<sub>21</sub>ON. MW, 183. Cryst. from Et<sub>2</sub>O. M.p. 68–9°.

v. Braun, Heymons, *Ber.*, 1928, 61, 2280.

### Norleucine (1-Amino-n-caproic acid)



C<sub>6</sub>H<sub>13</sub>O<sub>2</sub>N MW, 131

*d.*

Leaflets from H<sub>2</sub>O. M.p. 301°. Sol. to 2% in H<sub>2</sub>O at 25°. Very spar. sol. EtOH. [α]<sub>D</sub><sup>20</sup> + 23.14° in HCl, + 6.26° in H<sub>2</sub>O. Sweet taste. Sublimes partially at 275–80°.

*Cu salt*: dark blue needles from H<sub>2</sub>O. Blackens about 255°. Insol. EtOH.

N-Formyl: needles from H<sub>2</sub>O. M.p. 115–16°. [α]<sub>D</sub><sup>20</sup> – 15.85° in H<sub>2</sub>O. Very sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

N-Chloroacetyl: cryst. from AcOEt. M.p. 104–6°. [α]<sub>D</sub><sup>20</sup> + 3.56° in H<sub>2</sub>O.

N-Glycyl: prisms from EtOH.Aq. M.p. 239–40° decomp. [α]<sub>D</sub><sup>20</sup> – 8.71° in H<sub>2</sub>O.

N-Benzoyl: m.p. 53°.

*l.*

Leaflets from H<sub>2</sub>O. M.p. 301°. Sol. 60 parts H<sub>2</sub>O at 18°. [α]<sub>D</sub><sup>20</sup> – 26.5° in HCl, – 4.49° in

H<sub>2</sub>O. Sublimes partially at 275–80°. Bitter taste.

*N-Formyl*: needles from H<sub>2</sub>O. M.p. 115–16°.  $[\alpha]_D^{20} + 15.53^\circ$  in H<sub>2</sub>O. Very sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

*N-Glycyl*: prisms. M.p. 239–40° decomp.  $[\alpha]_D^{20} + 8.24^\circ$  in H<sub>2</sub>O.

*N-β-Naphthalenesulphonyl*: m.p. 149°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.  $[\alpha]_D - 22.54^\circ$ .

*N-Benzoyl*: m.p. 53°.

*dl.*

Leaflets from H<sub>2</sub>O. M.p. 297–300° (275°) (sealed tube). Sol. 83 parts H<sub>2</sub>O at 22°.

*Et ester*: C<sub>8</sub>H<sub>17</sub>O<sub>2</sub>N. MW, 159. Oil. B.p. 90–1°/11 mm.  $D^{17} 0.9335$ . *Picrate*: prisms from H<sub>2</sub>O. M.p. 124°.

*N-Me*: C<sub>7</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 145. Needles from H<sub>2</sub>O, leaflets from EtOH. Sublimes at 110°. Sol. 9.8 parts H<sub>2</sub>O at 11°, 43.7 parts 94% EtOH at 13°. Insol. Et<sub>2</sub>O. Bitter taste. FeCl<sub>3</sub> → intense red col.

*N-Di-Me*: C<sub>8</sub>H<sub>17</sub>O<sub>2</sub>N. MW, 159. Needles + 2H<sub>2</sub>O from H<sub>2</sub>O, needles from AcOEt. M.p. anhyd. 161–2°. Sol. EtOH, CHCl<sub>3</sub>. Mod. sol. warm C<sub>6</sub>H<sub>6</sub>. Spar. sol. AcOEt. Insol. Et<sub>2</sub>O, pet. ether. *Methoaurichloride*: yellow leaflets from EtOH. M.p. 142° (137–8°). *Methochloroplatinate*: m.p. 215–16°.

*N-Et*: C<sub>8</sub>H<sub>17</sub>O<sub>2</sub>N. MW, 159. Leaflets from EtOH. Sublimes without melting. Sol. 9.3 parts H<sub>2</sub>O at 15°, 63.5 parts 94% EtOH at 13°. Insol. Et<sub>2</sub>O. FeCl<sub>3</sub> → intense red col.

*N-Di-Et*: C<sub>10</sub>H<sub>21</sub>O<sub>2</sub>N. MW, 187. Cryst. from H<sub>2</sub>O or EtOH. Very sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

*N-Formyl*: needles from H<sub>2</sub>O. M.p. 114.5°. Sol. EtOH, Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O, AcOEt, C<sub>6</sub>H<sub>6</sub>, pet. ether.

*N-Chloroacetyl*: prisms from Me<sub>2</sub>CO.Aq. M.p. 104–7°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Insol. H<sub>2</sub>O.

*N-Glycyl*: leaflets or prisms from H<sub>2</sub>O. M.p. 210–15° decomp. Spar. sol. cold H<sub>2</sub>O.

Fischer, Hagenbach, *Ber.*, 1901, **34**, 3764.

Duvillier, *Bull. soc. chim.*, 1895, **13**, 484.

Abderhalden, Weil, *Z. physiol. Chem.*, 1912, **81**, 213.

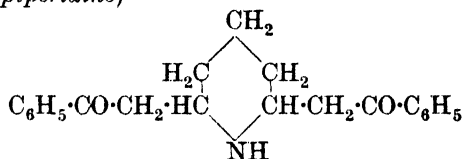
Abderhalden, Froehlich, Fuchs, *Z. physiol. Chem.*, 1913, **86**, 460.

Yaginuma, Hayakawa, Arai, *Chem. Abstracts*, 1932, **26**, 5073.

Abderhalden, Beckmann, *Z. physiol. Chem.*, 1932, **207**, 93.

Marvel, du Vigneaud, *Organic Syntheses*, Collective Vol. I, 40.

**Norlobelanine** (*Isolobelanine*, 2:6-diphenacylpiperidine)



C<sub>21</sub>H<sub>23</sub>O<sub>2</sub>N

MW, 321

One of lobelia alkaloids. Prisms from Et<sub>2</sub>O or EtOH.Aq. M.p. 120–1°. Sol. usual solvents. Optically inactive. Red. → norlobelanidine.

*B,HNO<sub>3</sub>*: prisms from EtOH.Aq. M.p. 193° decomp.

*B,HCl*: cryst. from EtOH. M.p. 201–2°.

*Methiodide*: prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 183–4°. Turns yellow in air.

*Benzoyl*: needles from EtOH. M.p. 125–6°.

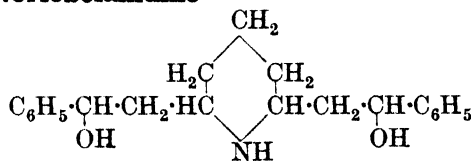
*N-Me*: Lobelanine, *q.v.*

Wieland, Hermesen, *Ann.*, 1926, **444**, 63.

Wieland, Koschkara, *Ann.*, 1929, **473**, 122.

Wieland, Drishaus, *ibid.*, 117.

**Norlobelanidine**



C<sub>21</sub>H<sub>27</sub>O<sub>2</sub>N

MW, 325

One of lobelia alkaloids. Needles from Et<sub>2</sub>O. M.p. 120°. Optically inactive.

*B,HCl*: needles from 80–90% EtOH. M.p. 244° slight decomp.

*B,HNO<sub>3</sub>*: prisms from H<sub>2</sub>O. M.p. 179–80°.

*Iodide*: needles from Me<sub>2</sub>CO.Aq. M.p. 211°.

*N-Me*: Lobelanidine, *q.v.*

Wieland, Koschkara, *Ann.*, 1929, **473**, 123.

Wieland, Drishaus, *ibid.*, 116.

**Normenthane.**

Isopropylcyclohexane, *q.v.*

**Nor-metahemipinic Acid.**

See 4:5-Dihydroxyphthalic Acid.

**Normorphine**

C<sub>16</sub>H<sub>17</sub>O<sub>3</sub>N

MW, 271

Cryst. M.p. 263–4°.

*B,HCl*: cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 305° decomp.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: cryst. + 3H<sub>2</sub>O. M.p. 230–1°.

*Me ether*: see Norcodeine.



*O-Isoamyl*:  $C_{21}H_{27}O_3N$ . MW, 341. Cryst. M.p.  $100^\circ$ . *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p.  $278^\circ$ . Spar. sol. H<sub>2</sub>O. *N-Cyano*: cryst. from EtOH. M.p.  $225^\circ$ . *N-Nitroso*: m.p.  $186^\circ$ .

*N-Cyano*: cryst. from EtOH. M.p.  $295-6^\circ$ . Mod. sol. CHCl<sub>3</sub>. Spar. sol. EtOH, Et<sub>2</sub>O, pet. ether.

*N-Nitroso*: needles from EtOH. M.p.  $267^\circ$  decomp. *Diacyl*: cryst. from EtOH.Aq. M.p.  $202-3^\circ$ .

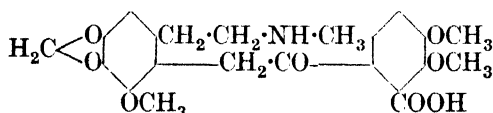
*Triacyl*: plates from EtOH.Aq. M.p.  $164^\circ$ .

*Dibenzoyl*: leaflets from EtOH. M.p.  $208^\circ$ . Sol. EtOH.

*N-Me*: Morphine, *q.v.*

Speyer, Walther, *Ber.*, 1930, **63**, 853.  
v. Braun, *Ber.*, 1914, **47**, 2320.

### Nornarceine



$C_{22}H_{25}O_8N$  MW, 431

Prisms from EtOH, needles + 3H<sub>2</sub>O from H<sub>2</sub>O. M.p. about  $205-22^\circ$  decomp., anhyd.  $229^\circ$  decomp. Sol. boiling EtOH.

*N-Me*: Narceine, *q.v.*

*Oxime*: leaflets from EtOH.Aq. M.p.  $171^\circ$ . Sol. H<sub>2</sub>O. Spar. sol. EtOH.

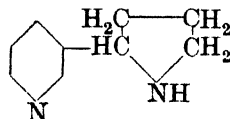
*B.HCl*: m.p.  $144^\circ$ .

Rabe, *Ber.*, 1907, **40**, 3283.

Rabe, McMillan, *Ber.*, 1910, **43**, 801.

Polonovski, Polonovski, *Bull. soc. chim.*, 1930, **47**, 365.

### Nornicotine (2-[3-Pyridyl]-pyrrolidine)



$C_9H_{12}N_2$  MW, 148

*d.*

Found in *Duboisia Hopwoodii*. B.p.  $117/3-6$  mm.  $D_4^{20}$  1.0757.  $n_D^{18.5}$  1.5490.  $[\alpha]_D^{20} + 86.3^\circ$ .

*Dipicrate*: m.p.  $191-2^\circ$ .

*Dipicrolonate*: m.p.  $252-3^\circ$ .

*l.*

Found in tobacco. B.p.  $130.5-131.3/11$  mm.,  $120/1$  mm.,  $D^{18.5}$  1.0737.  $n_D^{18.5}$  1.5378.  $[\alpha]_D^{22} - 88.8^\circ$ . Methylation  $\rightarrow$  *l*-nicotine.

*Chloroaurate*: m.p.  $217^\circ$ .

*Chloroplatinate*: decomp. at  $295^\circ$ .

*Dipicrate*: m.p.  $191-2^\circ$ .

*Dipicrolonate*: m.p.  $250-2^\circ$ .

*N-Phenylthiourethane*: m.p.  $176-7^\circ$ .

*N-Me*: see Nicotine.

*N-Et*:  $C_{11}H_{14}N_2$ . MW, 176. Oil. B.p.  $127-8/12$  mm. *Chloroaurate*: decomp. at  $203^\circ$ . *Picrate*: m.p.  $174-6^\circ$ .

*N-Allyl*:  $C_{12}H_{14}N_2$ . MW, 188. Liq. B.p.  $136-7/12$  mm. *Chloroaurate*: decomp. at  $145-8^\circ$ . *Chloroplatinate*: decomp. at  $255^\circ$ . *Picrate*: cryst. from EtOH. M.p.  $180-2^\circ$ .

*N-Acetyl*: b.p.  $212-14/12$  mm.  $[\alpha]_D^{20} - 3.24^\circ$  in C<sub>6</sub>H<sub>6</sub>. *Chloroplatinate*: decomp. at  $245^\circ$ . *Methiodide*: m.p.  $201^\circ$ . *Picrate*: m.p.  $151^\circ$ .

*N-Nitroso*: yellow liq. B.p.  $190-2^\circ/0.5$  mm. Misc. with H<sub>2</sub>O.  $B_2H_2PtCl_6$ : m.p.  $190^\circ$ . *Methiodide*: m.p.  $144^\circ$ . Hygroscopic.

*N-Urethane*: yellow cryst. from CHCl<sub>3</sub>-pet. ether. M.p.  $164-6^\circ$ .

v. Braun, Weissbach, *Ber.*, 1930, **63**, 2022.

Späth, Zajic, *Ber.*, 1935, **68**, 1667.

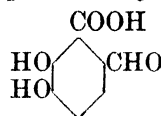
Späth, Hicks, Zajic, *Ber.*, 1935, **68**, 1388;  
1936, **69**, 250.

Späth, Marion, Zajic, *Ber.*, 1936, **69**, 251.

### Nornicotyrine.

See 2-[3-Pyridyl]-pyrrole.

**Nor-opianic Acid** (5:6-Dihydroxy-o-aldehydobenzoic acid, 6-formyl-o-pyrocatechuic acid, 5:6-dihydroxy-o-phthalaldehydic acid)



$C_8H_6O_5$  MW, 182

Cryst. +  $1\frac{1}{2}$ H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd.  $171^\circ$ . FeCl<sub>3</sub>  $\rightarrow$  deep bluish-green col.

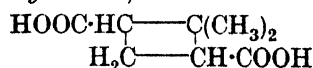
*5-Me ether*:  $C_9H_8O_5$ . MW, 196. Cryst. +  $2\frac{1}{2}$ H<sub>2</sub>O. M.p. anhyd.  $155-6^\circ$ . FeCl<sub>3</sub>  $\rightarrow$  dark blue col. *ψ-Me ester*.  $C_{10}H_{10}O_5$ . MW, 210. M.p.  $67-71^\circ$ . *ψ-Et ester*.  $C_{11}H_{12}O_5$ . MW, 224. Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p.  $104-6^\circ$ .

*5:6-Di-Me ether*: see Opianic Acid.

Wright, *J. Chem. Soc.*, 1877, **32**, 546.

Liebermann, *Ber.*, 1896, **29**, 2033; 1897, **30**, 692.

**Norpinic Acid** (1:1-Dimethylcyclobutane-2:4-dicarboxylic acid)



$C_8H_{12}O_4$  MW, 172

*Cis*:

Prisms from H<sub>2</sub>O. M.p.  $175^\circ$ . Sol. AcOEt, hot H<sub>2</sub>O. Mod. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Sublimes in needles above  $100^\circ$ .

*Di-Me ester*:  $C_{10}H_{16}O_4$ . MW, 200. B.p. 228–9°/756 mm., 113–113.8°/11 mm.  $D_4^{25}$  1.0700.  $n_D^{25}$  1.4459.

*Di-Et ester*:  $C_{12}H_{20}O_4$ . MW, 228. B.p. 140°/20 mm.

*Anhydride*:  $C_8H_{10}O_3$ . MW, 154. Plates from  $Et_2O$ . M.p. 135°. Warm  $H_2O \rightarrow$  *cis*-norpinic acid.

*Anilide*: m.p. 212–13°.

*Trans*:

Prisms from  $H_2O$ . M.p. 146°. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $Me_2CO$ .

Guha, Gaiind, *Chem. Abstracts*, 1934, **28**, 6721.

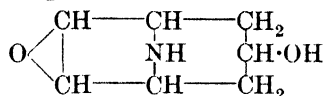
Kerr, *J. Am. Chem. Soc.*, 1929, **51**, 614.

Östling, *J. Chem. Soc.*, 1912, **101**, 475.

Perkin, Simonsen, *J. Chem. Soc.*, 1909, **95**, 1176.

Kerschbaum, *Ber.*, 1900, **33**, 891.

### Nor- $\psi$ -scopine



$C_7H_{11}O_2N$

MW, 141

Cryst. M.p. 184°. Excess methyl iodide  $\rightarrow$   $\psi$ -scopine methiodide.

*B,HCl*: m.p. 262°.

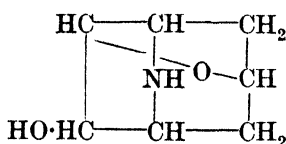
*B,HAuCl<sub>4</sub>*: m.p. 220°.

*Picrate*: m.p. 225°.

*N-Me*: see under Scopine.

Polonovski, Polonovski, *Compt. rend.*, 1928, **186**, 149.

### Norscopoline (Scopoligenin)



$C_7H_{11}O_2N$

MW, 141

Prisms from EtOH,  $Et_2O$ ,  $CHCl_3$  or pet. ether. M.p. 205–6°. Sol.  $H_2O$ , EtOH,  $CHCl_3$ . Spar. sol.  $Et_2O$ , pet. ether. Sublimes in needles at 120°. Methyl iodide  $\rightarrow$  scopoline.

*B,HCl*: m.p. 280°.

*B,HAuCl<sub>4</sub>*: golden-yellow cryst. M.p. 242° (236°).

*N-Me*: see Scopoline.

Luboldt, *Arch. Pharm.*, 1898, **236**, 22.

Hess, Merck, Uibrig, *Ber.*, 1915, **48**, 1906.

Polonovski, Polonovski, *Bull. soc. chim.*, 1927, **41**, 1206.

### Nortricycloekasantal.

See Norecsantalol.

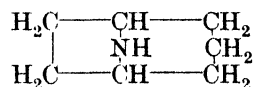
### Nortricycloekasantalic Acid.

See Norecsantallic Acid.

### Nortricycloekasantalol.

See Norecsantalol.

### Nortropene (2 : 5-Trimethylene-pyrrolidine)



$C_7H_{13}N$

MW, 111

Cryst. M.p. about 60°. B.p. about 161°. Sol.  $H_2O$ , EtOH,  $Et_2O$ . Absorbs  $CO_2$  from the air.

*B,HCl*: cryst. M.p. 281° decomp. Very sol.  $H_2O$ , EtOH.

*B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub>*: golden-yellow prisms. Decomp. at 225°.

*N-Phenylguanyl*: needles from EtOH.Aq. M.p. 145°. *B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub>*: decomp. at 208°. Insol.  $H_2O$ . *Picrate*: m.p. 157–8°.

*N-Benzoyl*: cryst. M.p. 94–5°. B.p. 204–5°/14 mm.

*N-Hydrocinnamoyl*: thick yellowish oil. B.p. 176–8°/0.4 mm.

Ladenburg, *Ber.*, 1887, **20**, 1649.

v. Braun, *Chem. Zentr.*, 1909, **II**, 1993; *Ber.*, 1911, **44**, 1259.

Hess, *Ber.*, 1918, **51**, 1014.

v. Braun, Weissbach, *Ber.*, 1930, **63**, 496.

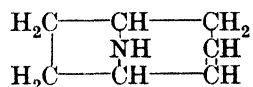
### Nortropanol.

See Nortropine.

### Nortropanone-3.

See Nortropinone.

### Nortropene (Nortropidine)



$C_7H_{11}N$

MW, 109

*B,HCl*: cryst.

*B,HAuCl<sub>4</sub>*: cryst. M.p. 187°.

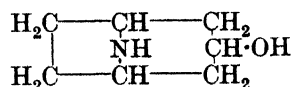
*N-Acetyl*: liq. Very sol.  $H_2O$ . Reacts neutral.

Polonovski, Polonovski, *Bull. soc. chim.*, 1927, **41**, 1202.

### Nortropidine.

See Nortropene.

### Nortropine (Nortropanol, tropigenin)



$C_7H_{13}ON$

MW, 127

Needles. M.p. 169° (161°). B.p. 233°. Sol. H<sub>2</sub>O, EtOH. Less sol. Et<sub>2</sub>O.

*B,HCl*: m.p. 285°.

*B,HNO<sub>2</sub>*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 160°. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

*B,HAuCl<sub>4</sub>*: golden-yellow leaflets from H<sub>2</sub>O. M.p. 215-16° decomp. Sol. EtOH.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: plates. M.p. 247° decomp. Very sol. H<sub>2</sub>O. Insol. EtOH.

*N-Acetyl*: cryst. from EtOH. M.p. 124°. *B,HCl*: m.p. 162°. Spar. sol. Me<sub>2</sub>CO.

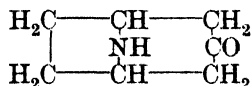
Polonovski, Polonovski, *Bull. soc. chim.*, 1927, 41, 1203.

Willstätter, *Ber.*, 1896, 29, 1579.

Merling, *Ann.*, 1883, 216, 343.

Chem. Werke Grenzach, D.R.P., 301,870, (*Chem. Zentr.*, 1918, I, 250).

**Nortropinone** (*Nortropanone-3*, 3-ketonortropane)



C<sub>7</sub>H<sub>11</sub>ON

MW, 125

Needles and leaflets from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 69-70°. Very sol. H<sub>2</sub>O, EtOH, C<sub>6</sub>H<sub>6</sub>. Sol. Et<sub>2</sub>O. Spar. sol. ligroin. Hygroscopic. Volatile in steam. Reacts strongly alkaline.

*B,HCl*: needles from EtOH. M.p. 201° decomp. Very sol. H<sub>2</sub>O. Insol. Et<sub>2</sub>O. Hygroscopic.

*Oxime*: leaflets from H<sub>2</sub>O. M.p. 181-2°. Very sol. hot H<sub>2</sub>O. Sol. EtOH. Spar. sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOEt, C<sub>6</sub>H<sub>6</sub>.

*B,HAuCl<sub>4</sub>*: golden-yellow prisms from EtOH. M.p. 168° decomp. Very sol. hot EtOH. Decomp. on heating with H<sub>2</sub>O.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-red prisms from H<sub>2</sub>O. Decomp. above 200°. Sol. hot H<sub>2</sub>O. Insol. EtOH.

*Picrate*: pale yellow prisms. M.p. 159-60°. Very sol. hot H<sub>2</sub>O. Sol. boiling EtOH.

Willstätter, *Ber.*, 1896, 29, 1581; D.R.P., 89,999, (*Chem. Zentr.*, 1897, I, 352).

Polonovski, Polonovski, *Bull. soc. chim.*, 1927, 41, 1202.

**Norvaline** (1-Amino-n-valeric acid)



C<sub>5</sub>H<sub>11</sub>O<sub>2</sub>N

MW, 117

*d.*

Cryst. from H<sub>2</sub>O. Sinters at 305°. [ $\alpha$ ]<sub>D</sub><sup>19</sup> - 21.84° in 20% HCl.

*Et ester*: C<sub>7</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 145. Liq. B.p.

77.5°/10 mm. *N-Benzoyl*: needles from ligroin. M.p. 59°. [ $\alpha$ ]<sub>D</sub><sup>19</sup> + 7.98°.

*N-Formyl*: cryst. M.p. 137°. [ $\alpha$ ]<sub>D</sub><sup>18</sup> + 2.05° in HCl. Spar. sol. H<sub>2</sub>O, EtOH, AcOEt. Insol. CHCl<sub>3</sub>, pet. ether.

*N-Chloroacetyl*: prisms. M.p. 107°. Sol. most ord. solvents. Spar. sol. pet. ether.

*N-Bromoacetyl*: m.p. 95°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> - 8.6° in EtOH.

*N-l- $\alpha$ -Bromopropionyl*: m.p. 105°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> - 18° in H<sub>2</sub>O.

*N-d- $\alpha$ -Bromopropionyl*: [ $\alpha$ ]<sub>D</sub><sup>22</sup> + 8.5° in EtOH. Very hygroscopic.

*N-Glycyl*: micro-prisms. Sinters at 223°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> - 10-17° in H<sub>2</sub>O. Sol. H<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>. Insol. EtOH, Et<sub>2</sub>O, AcOEt, pet. ether.

*N-Benzoyl*: cryst. + H<sub>2</sub>O from 20% EtOH. M.p. 64°, anhyd. 97°. [ $\alpha$ ]<sub>D</sub><sup>19</sup> - 15.0° in aq. alkalis.

*Brucine salt*: needles from EtOH. M.p. 143°.

*l.*

Cryst. from H<sub>2</sub>O. Sinters at 307°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 24.2° in 20% HCl.

*N-Formyl*: cryst. M.p. 136°. [ $\alpha$ ]<sub>D</sub><sup>18</sup> - 2.10° in EtOH.

*N-Chloroacetyl*: m.p. 108°.

*N-Bromoacetyl*: cryst. from EtOH-pet. ether. M.p. 92°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 9.2° in EtOH.

*N-l- $\alpha$ -Bromopropionyl*: prisms from CHCl<sub>3</sub>-pet. ether. M.p. 110°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> - 9.0° in EtOH.

*N-d- $\alpha$ -Bromopropionyl*: [ $\alpha$ ]<sub>D</sub><sup>22</sup> + 15° in H<sub>2</sub>O.

*N-Glycyl*: micro-prisms. Sinters at 220°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 10.28° in EtOH.

*N-Benzoyl*: needles + H<sub>2</sub>O from 20% EtOH. M.p. 64°, anhyd. 95°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 14.0° in aq. alkalis.

*dl.*

Silvery leaflets. M.p. 303° (291°) (sealed tube). Sol. hot H<sub>2</sub>O. Sol. 10 parts H<sub>2</sub>O at 18°. Insol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOEt, pet. ether. Aq. sol. has sweet taste.

*B,HCl*: prisms. M.p. 188° decomp. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

*B,HNO<sub>3</sub>*: prisms or plates. Sol. H<sub>2</sub>O. Less sol. EtOH. Insol. Et<sub>2</sub>O.

*Et ester*: cryst. M.p. 65°. B.p. 68.5°/8 mm. D<sub>4</sub><sup>15</sup> 0.9447. *N-Di-Et*: C<sub>11</sub>H<sub>23</sub>O<sub>2</sub>N. MW, 201. Oil. B.p. 80-5°/10-11 mm. *Picrate*: m.p. 116°.

*N-Me*: C<sub>6</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 131. Needles + H<sub>2</sub>O from H<sub>2</sub>O or EtOH. Aq., cryst. from MeOH. Sublimes at 252°. Sol. cold H<sub>2</sub>O, boiling EtOH. Sweet taste.

*N-Di-Me*: C<sub>7</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 145. Needles from AcOEt. M.p. 182°. Sol. H<sub>2</sub>O, EtOH.

Spar. sol. AcOEt. Insol. Et<sub>2</sub>O, pet. ether.  
*Nitrile*: C<sub>7</sub>H<sub>14</sub>N<sub>2</sub>. MW, 126. Liq. B.p. 175–6°. *Methiodide*: prisms from H<sub>2</sub>O. M.p. 181–2°. *Methochloroaurate*: yellow plates. M.p. 173–4° (160°). *Methochloroplatinate*: orange-yellow prisms. M.p. 219°.

*N-Formyl*: leaflets from EtOH. M.p. 132°. Sol. EtOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, AcOEt. Insol. CHCl<sub>3</sub>, pet. ether.

Cocker, Lapworth, *J. Chem. Soc.*, 1931, 1399.

Abderhalden, Schweitzer, *Chem. Zentr.*, 1931, I, 798.

Karrer, Schneider, *Helv. Chim. Acta*, 1930, 13, 1288.

Curtius, Lehmann, *J. prakt. Chem.*, 1930, 125, 228.

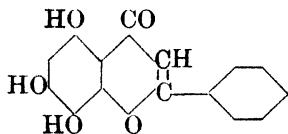
Abderhalden, Kürten, *Chem. Zentr.*, 1921, III, 296.

Menozi, Belloni, *Gazz. chim. ital.*, 1887, 17, 116.

Friedmann, *Beiträge zur Chemischen Physiologie und Pathologie*, 1908, 11, 170.

Schmidt, Dieterle, *Ann.*, 1910, 377, 48, 51. Slimmer, *Ber.*, 1902, 35, 404.

### Norwogonin (5 : 7 : 8-Trihydroxyflavone)



C<sub>15</sub>H<sub>10</sub>O<sub>5</sub>

MW, 270

Yellow needles from AcOH.Aq. M.p. 227–8°.

*Triacetyl*: needles from EtOH. M.p. 216–17°.

7 : 8-*Di-Me-5-Et ether*: C<sub>19</sub>H<sub>18</sub>O<sub>5</sub>. MW, 326. Needles from EtOH. M.p. 182–3°. Gives no col. with FeCl<sub>3</sub>.

*Tri-Me ether*: C<sub>18</sub>H<sub>16</sub>O<sub>5</sub>. MW, 312. Fine needles from H<sub>2</sub>O. M.p. 167–8°. Gives no col. with FeCl<sub>3</sub>. Insol. EtOH.Aq.

Hattori, *J. Chem. Soc. Japan*, 1930, 51, 472; *Acta Phytochimica*, 1931, 5, 219; 1932, 6, 177.

See also Nierenstein, *Acta Phytochimica*, 1932, 6, 173.

### Nostal.

See Noctal.

### Novain.

See Carnitine.

### Novarsenobenzene.

See Neosalvarsan.

### Novatophan.

See under 6-Methyl-2-phenylquinoline-4-carboxylic Acid.

**Novocaine** (*Ethocaine*, β-diethylaminoethyl p-aminobenzoate, *Procaine*)



C<sub>13</sub>H<sub>20</sub>O<sub>2</sub>N<sub>2</sub>

MW, 236

Needles + 2H<sub>2</sub>O from EtOH.Aq., plates from Et<sub>2</sub>O or ligroin. M.p. 51°, anhyd. 61°. Spar. sol. H<sub>2</sub>O. Used extensively as local anæsthetic.

*B,HCl*: needles. M.p. 153–6°. Very sol. H<sub>2</sub>O. Sol. 0.6 parts H<sub>2</sub>O, 30 parts EtOH at 25°. Spar. sol. CHCl<sub>3</sub>. Almost insol. Et<sub>2</sub>O. Decolourises acid KMnO<sub>4</sub>.

*B,HI*: m.p. 121–2°.

*B,HNO<sub>3</sub>*: cryst. M.p. 100–2°. Sol. H<sub>2</sub>O, EtOH.

*B,HClO<sub>3</sub>*: m.p. 89°.

*Borate*: m.p. 168° (159–60°).

*Picrate*: m.p. 146–7°.

*B,p-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·COOH*: cryst. from EtOH. M.p. 104°.

Einhorn, Uhlfelder, *Ann.*, 1909, 371, 136.

Dow Chemical Co., U.S.P., 1,501,635, (*Chem. Zentr.*, 1925, I, 901).

Hoffmann-La Roche Co., Swiss P., 118,336, (*Chem. Zentr.*, 1927, II, 977).

Jonesco-Matiu, Iliesco, *Chem. Zentr.*, 1936, I, 3339.

*Note*.—In view of the confusion existing in the literature regarding Ethocaine, Novocaine and Procaine due to differences between the British, German and American systems, all the data regarding the free base and its salts have been collected under the one name.

### Nucitol.

See under Inositol.

### Nuclidine.

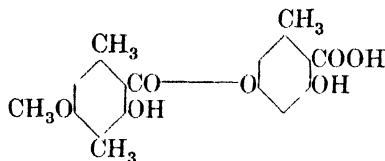
See Quinuclidine.

### NW-Acid.

See 1-Naphthol-4-sulphonic Acid.

## O

**Obtusatic Acid** (*Ramalic acid, protocetraric acid*)



$C_{18}H_{18}O_7$

MW, 346

Occurs in the lichen *Ramalina obtusata*, Arnold. Cryst. from  $Me_2CO$  or EtOH. M.p.  $203^\circ$  decomp. Mod. sol.  $Me_2CO$ . Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .  $FeCl_3 \rightarrow$  red col. with EtOH sol.

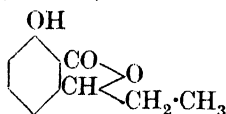
*Di-Me ether Me ester*:  $C_{21}H_{24}O_7$ . MW, 388. M.p.  $126^\circ$ .

*Diacetyl*: m.p.  $175^\circ$  decomp. *Me ester*: m.p.  $142^\circ$ .

Asahina, Fuzikawa, *Chem. Abstracts*, 1934, **28**, 2397; *Ber.*, 1932, **65**, 580.

Koller *et al.*, *Monatsh.*, 1932, **61**, 286; 1933, **62**, 241; 1934, **64**, 3.

**Ochracin** (*Mellein*)



$C_{10}H_{10}O_3$

MW, 178

Fermentation product of the mould *Aspergillus ochraceus*. M.p.  $58-58.5^\circ$ .  $FeCl_3 \rightarrow$  violet col.  $[\alpha]_D -124.86^\circ$  ( $[\alpha]_D^{25} -108.15^\circ$  in  $CHCl_3$ ).

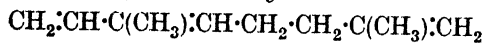
*Acetyl deriv.*: m.p.  $126-7^\circ$ .

*Benzoyl deriv.*: m.p.  $101-2^\circ$ .

*Me ether*:  $C_{11}H_{12}O_3$ . MW, 192. M.p.  $88-9^\circ$ .

Yabuta, Sumika, *British Chem. Abstracts*, 1935, **A**, 619.

**Ocimene** (2 : 6-Dimethyl-1 : 5 : 7-octatriene)



$C_{10}H_{16}$

MW, 136

Occurs in leaves of *Litsea Zeylanica*, C. & T., *Boronia dentigeroides*, Cheel, and *Ocimum basilicum*, L. B.p.  $176-8^\circ$  decomp.,  $81^\circ/30$  mm.,  $73-4^\circ/21$  mm.  $D_4^{21} 0.799$ .  $n_D^{25} 1.4857$ . Prolonged heating  $\rightarrow$  allo-ocimene (q.v.).

Romburg, *Chem. Zentr.*, 1901, **I**, 1006.

Penfold, *Chem. Abstracts*, 1929, **23**, 3303.

Enklaar, *Rec. trav. chim.*, 1926, **45**, 337.

**Octabromoacetylacetone** (*Phlorobromin, octabromopentandione-2 : 4*)



$C_5O_2Br_8$

MW, 732

Needles from  $CHCl_3$ . M.p.  $154-5^\circ$ . Mod. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , AcOH. Spar. sol. ligroin. Insol.  $H_2O$ .  $EtOH \rightarrow CBr_3 \cdot CO \cdot CHBr_2$ .  $NH_3 \rightarrow CBr_3 \cdot CO \cdot NH_2$ .

Benedikt, *Ann.*, 1877, **189**, 165.

Zincke, Kegel, *Ber.*, 1890, **23**, 1717.

**Octachloroacetylacetone** (*Octachloropentandione-2 : 4*)



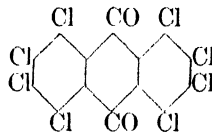
$C_5O_2Cl_8$

MW, 376

Needles or prisms from ligroin. M.p.  $42-3^\circ$ . B.p.  $165-8^\circ/30-2$  mm. Heat with  $H_2O \rightarrow CCl_3 \cdot COOH + CCl_3 \cdot CO \cdot CHCl_2$ .

Zincke, Kegel, *Ber.*, 1890, **23**, 240.

**Octachloroanthraquinone**



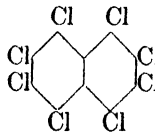
$C_{14}O_2Cl_8$

MW, 484

Yellow needles. Does not melt below  $360^\circ$ .

Eckert, *J. prakt. Chem.*, 1921, **102**, 361.

**Octachloronaphthalene**



$C_{10}Cl_8$

MW, 404

Cryst. from  $C_6H_6-CCl_4$ . M.p.  $197.5-198^\circ$ . B.p.  $440-2^\circ/7.4$  mm.,  $258-60^\circ/2.5$  mm.,  $246-50^\circ/0.5$  mm.

Schwemberger, Gordon, *Chem. Zentr.*, 1935, **II**, 514; *Chem. Abstracts*, 1933, **27**, 2439.

**Octacosane**



$C_{28}H_{58}$

MW, 394

M.p. 64-5° (62°). B.p. 278°/15 mm., 224°/1.1 mm.  $n_D^{25}$  1.43539.

Levene, West, Scheer, *J. Biol. Chem.*, 1915, **20**, 521.

Hildebrand, Wachter, *J. Am. Chem. Soc.*, 1929, **51**, 2487.

**Octacosane-1 : 28-dicarboxylic Acid**

$\text{C}_{30}\text{H}_{58}\text{O}_4$  MW, 482

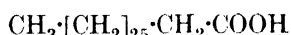
M.p. 108° (123-5°).

*Di-Et ester* :  $\text{C}_{34}\text{H}_{66}\text{O}_4$ . MW, 538. M.p. 74°.

Fairweather, *Chem. Abstracts*, 1927, **21**, 3182.

Ruzicka, Brugger, Seidel, Schinz, *Helv. Chim. Acta*, 1928, **11**, 496.

**Octacosanic Acid** (*Octacosanoic acid*, *octacosoic acid*)



$\text{C}_{28}\text{H}_{56}\text{O}_2$  MW, 424

M.p. 90.3-90.5° (89°).  $n_D^{100}$  1.4313.

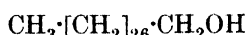
*Et ester* :  $\text{C}_{30}\text{H}_{60}\text{O}_2$ . MW, 452. M.p. 64.8-65°.

Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2512.

Holde, Bleyberg, Vohrer, *Chem. Abstracts*, 1931, **25**, 189.

**Octacosanol-1.**

*See* Octacosyl Alcohol.

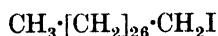
**Octacosyl Alcohol** (*Octacosanol*)

$\text{C}_{28}\text{H}_{58}\text{O}$  MW, 426

M.p. 83.2-83.4° (82.9-83.1°).

Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2512.

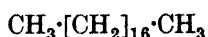
Pollard, Chibnall, Piper, *Biochem. J.*, 1933, **27**, 1889.

**Octacosyl iodide** (*1-Iodo-n-octacosane*)

$\text{C}_{28}\text{H}_{57}\text{I}$  MW, 520

M.p. 62.8-63.2°.

Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2512.

**Octadecane**

$\text{C}_{18}\text{H}_{38}$  MW, 254

F.p. 27.5°. (i) Transparent cryst. from  $\text{Me}_2\text{CO}$ . M.p. 27.6°. (ii) Opaque cryst. M.p.

28.02°. B.p. 305-7°, 185-7°/20 mm., 180-3°/15 mm.  $D_4^{25}$  0.7768.

Ueno, *Chem. Abstracts*, 1930, **24**, 4948.

Schrauth, Schenck, Stickdorn, *Ber.*, 1931, **64**, 1317.

Matsui, Arakawa, *Chem. Abstracts*, 1932, **26**, 5264.

Carey, Smith, *J. Chem. Soc.*, 1933, 346.

**Octadecane-1 : 18-dicarboxylic Acid**

$\text{C}_{20}\text{H}_{38}\text{O}_4$  MW, 342

M.p. 124-5° (123°).

*Di-Me ester* :  $\text{C}_{22}\text{H}_{42}\text{O}_4$ . MW, 370. M.p. 65.5-66°. B.p. 233-4°/2 mm.

*Di-Et ester* :  $\text{C}_{24}\text{H}_{46}\text{O}_4$ . MW, 398. M.p. 54.5-55°. B.p. 230-2°/2 mm.

Chuit, Hausser, *Helv. Chim. Acta*, 1929, **12**, 856.

**Octadecanol-1.**

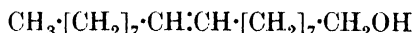
*See* Octadecyl Alcohol.

**Octadecanol-3.**

*See* Ethylpentadecylcarbinol.

**Octadecanone.**

*See* Ethyl pentadecyl Ketone and Methyl hexadecyl Ketone.

 **$\Delta^9$ -Octadecenyl Alcohol** (*9-Octadecenol-1*)

$\text{C}_{18}\text{H}_{36}\text{O}$  MW, 268

*Cis* : Oleyl alcohol, oleic alcohol.

Occurs in fish oils. B.p. 205-10°/15 mm.  $D_4^{20}$  0.8489.  $n_D^{20}$  1.4607. Conc. sulphuric acid  $\rightarrow$  sulphuric esters possessing powerful detergent, wetting, and emulsifying properties.

*Acetyl* :  $D_4^{20}$  0.8704.  $n_D^{20}$  1.4515.

Reid, Cockerille, Meyer, Cox, Ruhoff,

*Organic Syntheses*, 1935, **XV**, 72.

Noller, Bannerot, *J. Am. Chem. Soc.*, 1934, **56**, 1563.

Hirose, *Chem. Abstracts*, 1930, **24**, 2321.

Toyama, *Chem. Abstracts*, 1924, **18**, 1270.

*Trans* : elaidyl alcohol, elaidic alcohol.

Constituent of sperm oil. M.p. 36-7° (34°). B.p. 216°/18 mm.  $D_4^{20}$  0.8388.  $n_D^{20}$  1.4552.

*Phenylurethane* : m.p. 55°.

$\beta$ -Naphthylurethane : m.p. 71°.

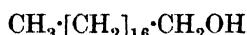
Böeseken, Belinfante, *Rec. trav. chim.*, 1926, **45**, 915.

André, Francois, *Compt. rend.*, 1927, **185**, 279.

Toyama, *Chem. Umschau*, 1924, **31**, 13.

**Octadecine-1.**

See Hexadecylacetylene.

**Octadecyl Alcohol** (1-Hydroxyoctadecane, stearyl alcohol, octadecanol-1)
 $\text{C}_{18}\text{H}_{38}\text{O}$  MW, 270

Leaflets from EtOH. F.p. 57-95°. M.p. 59-4-59-8°. B.p. 210°/15 mm., 195-205°/0-2 mm.  $D_4^{20}$  0-8124. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  sulphuric ester possessing detergent, wetting, and emulsifying properties.

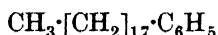
Acetyl: (i) f.p. 30-25. (ii) M.p. 32-85°.

Phenylurethane: needles from EtOH. M.p. 79-80°.

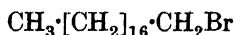
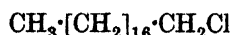
o-Nitrophenylurethane: m.p. 70°.

m-Nitrophenylurethane: m.p. 77°.

3:5-Dinitrophenylurethane: m.p. 88°.

Me ether:  $\text{C}_{19}\text{H}_{40}\text{O}$ . MW, 284. Laminæ from  $\text{Et}_2\text{O}$ -MeOH. M.p. 30-1°. $\alpha$ -Glyceryl ether: see Batyl Alcohol. $\beta$ -Glyceryl ether:  $\text{C}_{21}\text{H}_{44}\text{O}_3$ . MW, 344. Needles from EtOH. M.p. 62-3°. Di-phenylurethane: m.p. 83-4°.Jantzen, Tiedcke, *J. prakt. Chem.*, 1930, 127, 277.Schrauth, Schlenck, Stiekdorn, *Ber.*, 1931, 64, 1318.Bleyberg, Ulrich, *Ber.*, 1931, 64, 2510.Phillips, Mumford, *J. Chem. Soc.*, 1932, 904; 1933, 235.Tsujiimoto, *Chem. Abstracts*, 1921, 15, 2006.**Octadecylbenzene** (1-Phenyloctadecane)
 $\text{C}_{24}\text{H}_{42}$  MW, 330

M.p. 36°. B.p. 249°/15 mm., 147°/0 mm.

Krafft, *Ber.*, 1886, 19, 2984.**Octadecyl bromide** (1-Bromo-octadecane)
 $\text{C}_{18}\text{H}_{37}\text{Br}$  MW, 333
Cryst. from EtOH. M.p. 28-5°. Sol. EtOH,  $\text{AcOEt}$ , pet. ether.Oskerkko, *Chem. Zentr.*, 1914, II, 1264.**Octadecyl chloride** (1-Chloro-octadecane)
 $\text{C}_{18}\text{H}_{37}\text{Cl}$  MW, 288-5
B.p. 185-90°/15 mm.  $D_4^{20}$  0-9041.Mabery, *Am. Chem. J.*, 1902, 28, 165. **$\Delta^2$ -Octadecylenic Acid**
 $\text{C}_{18}\text{H}_{34}\text{O}_2$  MW, 282

Cryst. from AcOH. M.p. 56-7°.

Me ester:  $\text{C}_{19}\text{H}_{36}\text{O}_2$ . MW, 296. M.p. 36°.Eckert, Halla, *Monatsh.*, 1913, 34, 1815. **$\Delta^3$ -Octadecylenic Acid**
 $\text{C}_{18}\text{H}_{34}\text{O}_2$  MW, 282

M.p. 52-3°.

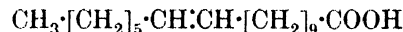
See previous reference.

 **$\Delta^5$ -Octadecylenic Acid.**

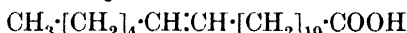
See Petroselic Acid.

 **$\Delta^9$ -Octadecylenic Acid.**

See Elaidic Acid and Oleic Acid.

 **$\Delta^{10}$ -Octadecylenic Acid**
 $\text{C}_{18}\text{H}_{34}\text{O}_2$  MW, 282

F.p. 6-8°.

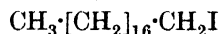
Fokin, *Chem. Abstracts*, 1912, 6, 2409. **$\Delta^{11}$ -Octadecylenic Acid**
 $\text{C}_{18}\text{H}_{34}\text{O}_2$  MW, 282

F.p. 36-8°. M.p. 34-6°.

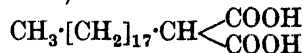
See previous reference.

**Octadecylic Acid.**

See Stearic Acid.

**Octadecyl iodide** (1-Iodo-octadecane)
 $\text{C}_{18}\text{H}_{37}\text{I}$  MW, 380

M.p. 34-5-35° (32-94°). B.p. 160-70°/0-5 mm. Spar. sol. EtOH.

Jantzen, Tiedcke, *J. prakt. Chem.*, 1930, 127, 277.Bleyberg, Ulrich, *Ber.*, 1931, 64, 2510.Carey, Smith, *J. Chem. Soc.*, 1933, 346.**Octadecylmalonic Acid** (Nonadecane-1:1-dicarboxylic acid)
 $\text{C}_{21}\text{H}_{40}\text{O}_4$  MW, 356
Cryst. from AcOH. M.p. 109-10°. Sol.  $\text{Et}_2\text{O}$ . Insol. pet. ether.Amide:  $\text{C}_{21}\text{H}_{41}\text{O}_3\text{N}$ . MW, 355. Cryst. from AcOH. M.p. 126° with loss of  $\text{CO}_2$ . Insol.  $\text{Et}_2\text{O}$ .

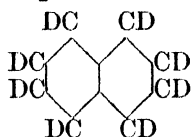
Mononitrile: 1-cyano-n-eicosanic acid.

$C_{21}H_{39}O_2N$ . MW, 337. Cryst. powder from EtOH. M.p. 88°.

Baczewski, *Monatsh.*, 1896, **17**, 544.

Meyer, Brod, Soyka, *Monatsh.*, 1913, **34**, 1132.

### Octadeuteronaphthalene



$C_{10}D_8$  MW, 136

Cryst. from MeOH.Aq. M.p. 77.5°.

Clemo, McQuillen, *J. Chem. Soc.*, 1935, 1325.

**2:4-Octadiene** (*Propylpropenylethylene, methylpropylbutadiene*)

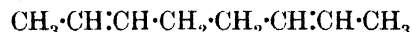


$C_8H_{14}$  MW, 110

B.p. 133.5–134°.  $D_4^{25}$  0.7427.  $n_D^{25}$  1.4542.

Mulliken, Wakeman, Gerry, *J. Am. Chem. Soc.*, 1935, **57**, 1607.

**2:6-Octadiene** (*Dicrotyl, propenylethylidene-propane*)

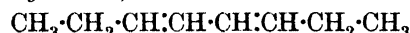


$C_8H_{14}$  MW, 110

B.p. 117–19°.

Charon, *Ann. chim.*, 1899, **17**, 265.

**3:5-Octadiene** (*1:4-Diethylbutadiene, dipropenylethane*)

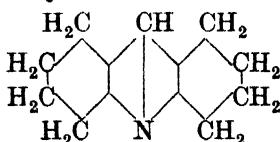


$C_8H_{14}$  MW, 110

B.p. 138–40°.

Kaufmann, Schweitzer, *Ber.*, 1922, **55**, 262.

### sym.-Octahydroacridine



$C_{13}H_{17}N$  MW, 187

Cryst. from ligroin. M.p. 69°. B.p. 175°/17 mm.

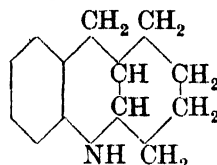
$B_2, H_2PtCl_6$ : m.p. 199–200°.

*Picrate*: m.p. 195°.

*Methiodide*: m.p. 159°.

Braun, Petzold, Schultheiss, *Ber.*, 1923, **56**, 1349.

### unsym.-Octahydroacridine



$C_{13}H_{17}N$

MW, 187

*d.*

(a) M.p. 84.5°.  $[\alpha]_D + 34^\circ$ . (b) M.p. 72°.  $[\alpha]_D + 22^\circ$ .

*l.*

(a) M.p. 85°.  $[\alpha]_D - 32^\circ$ . (b) M.p. 72°.  $[\alpha]_D - 24^\circ$ .

*Camphorsulphonate*: (a) M.p. 183°. (b) M.p. 172°.

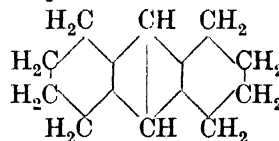
*dl.*

(a) M.p. 84°. B.p. 183°/12 mm. *N-Benzoyl*: m.p. 104°. (b) Plates from pet. ether. M.p. 72°. *N-Acetyl*: m.p. 136°. *Picrate*: m.p. 175°. *Methiodide*: m.p. 217°.

Braun, Heymons, Manz, *Ber.*, 1931, **64**, 233.

Perkin, Sedgwick, *J. Chem. Soc.*, 1924, 125, 2448.

### sym.-Octahydroanthracene



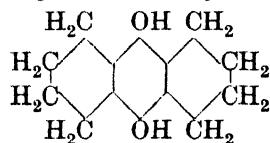
$C_{14}H_{18}$

MW, 186

Leaflets from EtOH. M.p. 39°.

Braun, Bayer, *Ber.*, 1925, **58**, 2679.

### sym.-Octahydroanthrahydroquinone



$C_{14}H_{18}O_2$

MW, 218

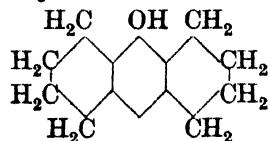
Cryst. from EtOH. M.p. 242° (234–6°).

*Diacetyl*: m.p. 224°.

Braun, Bayer, *Ber.*, 1925, **58**, 2680.

Skita, *ibid.*, 2692.

### sym.-Octahydroanthranol



$C_{14}H_{18}O$

MW, 202



**sym.-Octahydroanthraquinone**

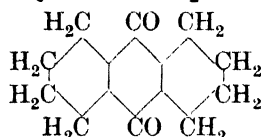
Needles from EtOH. M.p. 125°.

Acetyl : m.p. 52°.

Benzoyl : m.p. 129° (128°).

Skita, *Ber.*, 1925, **58**, 2693.

Braun, Bayer, *ibid.*, 2678.

**sym.-Octahydroanthraquinone**

$C_{14}H_{16}O_2$

MW, 216

*Cis-cis* :

Leaflets from MeOH. M.p. 154-5°.

*Cis-trans* :

Needles from EtOH. M.p. 186°.

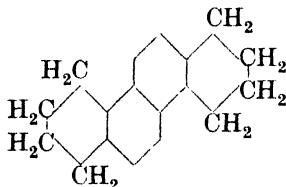
*Trans-trans* :

Leaflets from AcOEt. M.p. 245°.

Alder, Stein, *Ann.*, 1933, **501**, 283.

Skita, *Ber.*, 1925, **58**, 2692.

Braun, Bayer, *ibid.*, 2678.

**sym.-Octahydrochrysene**

$C_{18}H_{20}$

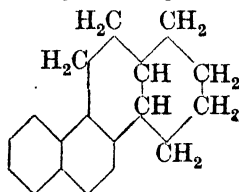
MW, 236

M.p. 136-8° (138-40°). B.p. 180-1°/0.2 mm.

Picrate : m.p. 139-40°.

Braun, Irmisch, *Ber.*, 1932, **65**, 885.

Cohen, Cook, Hewett, *J. Chem. Soc.*, 1935, 1636.

**unsym.-Octahydrochrysene**

$C_{18}H_{20}$

MW, 236

*Cis* (?) :

Needles from EtOH. M.p. 78-9°.

Picrate : m.p. 106-106.5°.

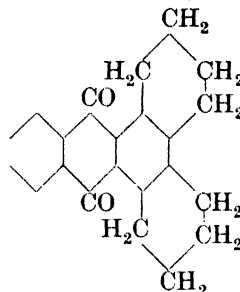
*Trans* (?) :

Needles from MeOH. M.p. 114-114.5°.

Forms no picrate.

Cook, Hewett, *J. Chem. Soc.*, 1934, 372.

304

**1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydro-naphthalene****Octahydro-1 : 2 : 3 : 4-dibenzanthraquinone**

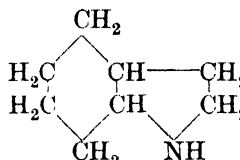
$C_{22}H_{20}O_2$

MW, 316

Yellow cryst. from xylene. M.p. 234°.

See previous reference and also

Barnett, Goodway, Lawrence, *J. Chem. Soc.*, 1935, 1684.

**Octahydroindole (Perhydroindole)**

$C_8H_{15}N$

MW, 125

Oil with onion-like odour. B.p. 185.5°/760 mm., 182-3°/720 mm., 64-5°/12 mm. Sol. usual org. solvents. Insol.  $H_2O$ . Reacts alkaline.  $D_4^{20}$  0.9472.  $n_D^{20}$  1.4892.

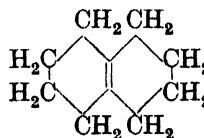
$B_2H_2PtCl_6$  : reddish-yellow leaflets from EtOH. M.p. about 172-3°. Very sol. warm EtOH. Insol. cold  $H_2O$ .

*Benzenesulphonyl* : cryst. M.p. 70-1°.

*Picrate* : needles from EtOH. M.p. 137-8°. Very sol. hot EtOH,  $CHCl_3$ . Spar. sol.  $C_6H_6$ .

Willstätter, Jaquet, *Ber.*, 1918, **51**, 778.

Willstätter, Seitz, Braun, *Ber.*, 1925, **58**, 385.

**1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydro-naphthalene ( $\Delta^9$ -Octalin)**

$C_{10}H_{16}$

MW, 136

B.p. 196.5-198.8°/759 mm.  $D_4^{20}$  0.9145.  $n_D^{20}$  1.4978.

*Nitroschloride* : m.p. 89-90°.

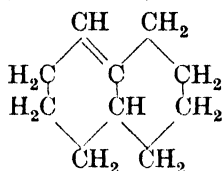
Hückel, Naab, *Ann.*, 1933, **502**, 144.

Schuilin, *Chem. Zentr.*, 1935, II, 3650.

Cf. Nametkin, Madaeff-Ssitscheff, *Ber.*, 1926, **59**, 373.

**1 : 2 : 3 : 4 : 5 : 6 : 7 : 10-Octahydro-naphthalene**

**1 : 2 : 3 : 4 : 5 : 6 : 7 : 10-Octahydro-naphthalene ( $\Delta^{1,9}$ -Octalin)**



$C_{10}H_{16}$

MW, 136

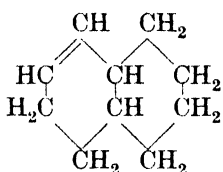
B.p. 189.5–193.5°/759 mm.  $D_4^{20}$  0.9027.

Nitropiperidide : m.p. 179° decomp.

Nitrosochloride : m.p. 127°.

See previous references.

**1 : 2 : 3 : 4 : 5 : 6 : 9 : 10-Octahydro-naphthalene ( $\Delta^1$ -Octalin)**



$C_{10}H_{16}$

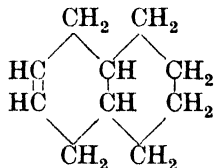
MW, 136

B.p. 185°.  $D_4^{16}$  0.8970.

Hückel, Naub, *Ann.*, 1933, 502, 150.

Cf. Borsche, Lange, *Ann.*, 1923, 434, 225.

**1 : 2 : 3 : 4 : 5 : 8 : 9 : 10-Octahydro-naphthalene ( $\Delta^2$ -Octalin,  $\beta$ -naphthanene)**



$C_{10}H_{16}$

MW, 136

B.p. 197–9°.  $D_4^{20}$  0.9103.  $n_D^{20}$  1.4941.

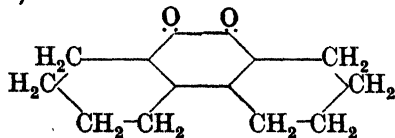
*Trans* :

M.p. — 24°.  $D_4^{20}$  0.8936.  $n_D^{20}$  1.4841.

Hückel, *Ber.*, 1925, 58, 1451.

Cf. Borsche, Lange, *Ann.*, 1923, 434, 225.

***sym.*-Octahydrophenanthraquinone (Oct-anthrone)**



$C_{14}H_{16}O_2$

MW, 216

Red needles from MeOH. M.p. 142°.

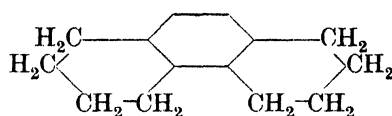
Skita, Warnat, *Ber.*, 1925, 58, 2691.

Dict. of Org. Comp.—III.

305

**1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-Octahydro-phenazine**

**1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydrophen-anthrene (Octanthrene)**



$C_{14}H_{18}$

MW, 186

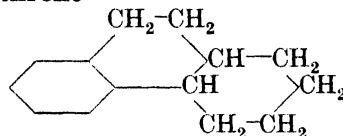
M.p. 16.7°. B.p. 295°, 167.5°/13 mm.  $D_4^{20}$  1.026.  $n_D^{17}$  1.5669.

Schroeter, Müller, Huang, *Ber.*, 1929, 62, 650.

Schroeter, D.R.P., 352,719, (*Chem. Abstracts*, 1923, 17, 1246).

Kamp, Mosettig, *J. Am. Chem. Soc.*, 1935, 57, 1107.

**1 : 2 : 3 : 4 : 9 : 10 : 11 : 12-Octahydro-phenanthrene**



$C_{14}H_{18}$

MW, 186

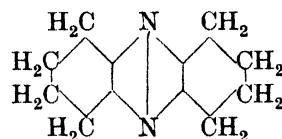
B.p. 159°/15 mm., 135°/9 mm. (135–7°/6.5 mm.).  $D_4^{22}$  0.997325.  $n_D^{19.2}$  1.5527.

Fulton, Robinson, *J. Chem. Soc.*, 1933, 1465.

Bergs, *Ber.*, 1934, 67, 243.

Kagehira, *Chem. Abstracts*, 1932, 26, 443.

**1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydrophen-azine**



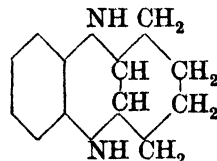
$C_{12}H_{16}N_2$

MW, 188

Cryst. from EtOH.Aq. M.p. 109°.

Clemo, McIlwain, *J. Chem. Soc.*, 1936, 258.

**1 : 2 : 3 : 4 : 9' : 10 : 11 : 12-Octahydro-phenazine**



$C_{12}H_{16}N_2$

MW, 188

(i) Needles from EtOH. M.p. 156°.

N : N' Di-nitroso : m.p. 126°.

(ii) Plates from EtOH. M.p. 147°. N : N'-Dinitroso : m.p. 109°.

See previous reference.

### Octahydropyrindole.

See Indolizidine.

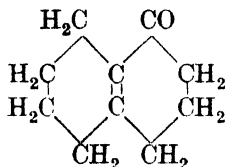
### n-Octaldehyde.

See Caprylic Aldehyde.

### Octalin.

See Octahydronaphthalene.

$\Delta^9$ - $^{10}$ -Octalone (1-Keto-octahydronaphthalene)



$C_{10}H_{14}O$

MW, 150

B.p. about 140°/9 mm.  $D_4^{20}$  1.000.  $n_D^{20}$  1.4996.

Osime : m.p. 144-5°. Benzoyl deriv. : m.p. 130.5-131.5°.

Semicarbazone : m.p. 242-3°.

Cook, Lawrence, *J. Chem. Soc.*, 1935, 1638.

### Octamethylene.

See Cyclo-octane.

**Octamethylenediamine** (1 : 8-Diamino-n-octane)



$C_8H_{20}N_2$

MW, 144

Plates. M.p. 52°. B.p. 240-1° (236-40°, 225-6°), 130-40°/20 mm. Sol.  $H_2O$ , EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ , ligroin.

$B_2HCl$  : m.p. 274°.

$B_2HAuCl_4$  : m.p. 188-9° decomp.

$B_2(COOH)_2$  : m.p. 223°.

Di-picrate : m.p. 180° decomp. (182-3°).

N : N'-Diacyetyl : m.p. 121-2°.

Naegeli, Lendorff, *Helv. Chim. Acta*, 1932, 15, 53.

Neuberg, Neimann, *Z. physiol. Chem.*, 1905, 45, 117.

**Octamethylene Glycol** (1 : 8-Dihydroxy-n-octane, octandiol-1 : 8)



$C_8H_{18}O_2$

MW, 146

Needles from  $C_6H_6$ -ligroin. M.p. 63° (60°). B.p. 172°/20 mm., 164°/12 mm., 160-2°/9.5 mm. Sol. EtOH. Spar. sol.  $H_2O$ ,  $Et_2O$ , ligroin.

Diphenylurethane : m.p. 172-172.5°.

Di-Me ether :  $C_{10}H_{22}O_2$ . MW, 174. B.p. 221°.  $D_4^{20}$  0.8613.  $n_D^{20}$  1.4257.

Di-n-amyl ether :  $C_{18}H_{38}O_2$ . MW, 286. B.p. 212°/35 mm.

Lespieau, *Compt. rend.*, 1914, 158, 1188.

Dionneau, *Bull. soc. chim.*, 1910, 7, 328.

Bouveault, Blanc, D.R.P., 164,294, (*Chem. Zentr.*, 1905, II, 1701).

### Octamethylsucrose.

See under Sucrose.

### Octanal.

See Caprylic Aldehyde.

**Octandiol-1 : 2** (1 : 2-Dihydroxy-n-octane, n-hexylethylene glycol)



$C_8H_{18}O_2$

MW, 146

Cryst. from pet. ether. M.p. 35-7°. B.p. 135-6°/20 mm.  $[\alpha]_D^{20}$  -4.7° in EtOH.

Späth, Kuffner, Ensfeßner, *Ber.*, 1933, 66, 598.

### Octandiol-1 : 8.

See Octamethylene Glycol.

**Octandiol-2 : 7** (2 : 7-Dihydroxy-n-octane)



$C_8H_{18}O_2$

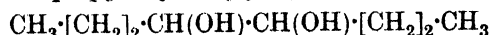
MW, 146

Oil. B.p. 138-9°/15 mm. Sol. ord. org. solvents.

Di-phenylurethane : m.p. 126°.

Blaise, Koehler, *Bull. soc. chim.*, 1910, 7, 417.

**Octandiol-4 : 5** (4 : 5-Dihydroxy-n-octane, sym.-dipropylethylene glycol)



$C_8H_{18}O_2$

MW, 146

Meso-.

M.p. 123-4°. B.p. 115-18°/10 mm.

Bouveault, Locquin, *Bull. soc. chim.*, 1906, 35, 644.

Veibel, *Biochem. Z.*, 1931, 239, 456.

### Octandione-2 : 3.

See Acetylcaproyl.

**Octandione-2 : 4** (Acetylvalerylmethane, 2 : 4-diketo-octane)



$C_8H_{14}O_2$

MW, 142

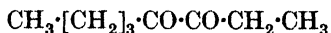
B.p. 64-6°/5 mm.  $D_4^{25}$  0.9218.

Kutz, Adkins, *J. Am. Chem. Soc.*, 1930, 52, 4042.

### Octandione-2 : 7.

See 1 : 4-Diacetobutane.

**Octandione-3 : 4** (*Propionylvaleryl*, 3 : 4-diketo-octane)



$\text{C}_8\text{H}_{14}\text{O}_2$  MW, 142

*Dioxime* : m.p. about 139–41°.

Fileti, Ponzio, *Gazz. chim. ital.*, 1898, **28**, ii, 264.

**Octandione-3 : 5** (*Propionylbutyrylmethane*, 3 : 5-diketo-octane)



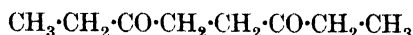
$\text{C}_8\text{H}_{14}\text{O}_2$  MW, 142

B.p. 189–90°, 75°/20 mm.

*Cu deriv.* : blue cryst. from EtOH. M.p. 158.5° (157°).

Powell, Seymour, *J. Am. Chem. Soc.*, 1931, **53**, 1050.

**Octandione-3 : 6** (sym.-*Dipropionylethane*, 3 : 6-diketo-octane)



$\text{C}_8\text{H}_{14}\text{O}_2$  MW, 142

Plates. M.p. 34–5°. B.p. 98°/14 mm.

*Dioxime* : m.p. 169°.

Blaise, *Compt. rend.*, 1914, **158**, 506.

**Octandione-4 : 5.**

See Dibutyryl.

**n-Octane** (1-Ethylhexane)



$\text{C}_8\text{H}_{18}$  MW, 114

F.p. — 56.82°. M.p. — 56.90°. B.p. 125.59° (125.3–125.6°).  $D_4^{20}$  0.7024 (0.70279).  $n_D^{20}$  1.39750 (1.39760).

Gardiner, Borgstrom, *J. Am. Chem. Soc.*, 1929, **51**, 3375.

Fischer, Klemm, *Z. physik. Chem.*, 1930, **A**, **147**, 275.

Shepard, Henne, Midgley, *J. Am. Chem. Soc.*, 1931, **53**, 1949.

Moldawski, Liwischitz, *Chem. Zentr.*, 1936, I, 758.

Waterman, Kok, *Rec. trav. chim.*, 1934, **53**, 725.

Mair, *Bureau of Standards Journal of Research*, 1932, **9**, 463.

Whitmore, Laughlin, *J. Am. Chem. Soc.*, 1933, **55**, 5056 (*Bibl.*).

Maman, *Chem. Zentr.*, 1936, I, 2332.

**Octane-1 : 8-dicarboxylic Acid.**

See Sebacic Acid.

**Octanol.**

See *n*-Octyl Alcohol, *sec.-n*-Octyl Alcohol, Ethyl-*n*-amylcarbinol, and Propylbutylcarbinol.

**Octanone.**

See Ethyl *n*-amyl Ketone, Methyl *n*-hexyl Ketone, and Propyl butyl Ketone.

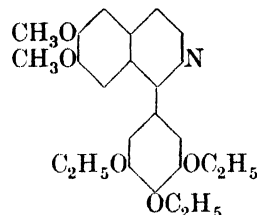
**Octanthrene.**

See 1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-Octahydrophenanthrene.

**Octanthrone.**

See Octahydrophenanthraquinone.

**Octaverine** (1-[3 : 4 : 5-Triethoxyphenyl]-6 : 7-dimethoxyisoquinoline)



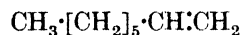
$\text{C}_{21}\text{H}_{23}\text{O}_3\text{N}$  MW, 337

*B, HCl* : m.p. 199–200°.

Ellinger, Koschara, Seegar, *Chem. Abstracts*, 1934, **28**, 4476.

Asta A.G., F.P., 760,825, (*Chem. Abstracts*, 1934, **28**, 4178).

**1-Octene** (1-Octylene)



$\text{C}_8\text{H}_{16}$  MW, 112

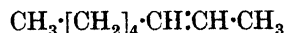
F.p. — 104° C. B.p. 121.85–122.15° (119–119.5°).  $D_4^{20}$  0.7155 (0.715).  $n_D^{20}$  1.40880 (1.4087).

Waterman, Kok, *Rec. trav. chim.*, 1934, **53**, 725 (*Bibl.*).

Whitmore, Herndon, *J. Am. Chem. Soc.*, 1933, **55**, 3429.

Bourguel, *Bull. soc. chim.*, 1927, **41**, 1476.

**2-Octene** (2-Octylene)



$\text{C}_8\text{H}_{16}$  MW, 112

B.p. 124.1–124.7°/745 mm. (123–123.5°).  $D_4^{20}$  0.7248.  $n_D^{20}$  1.4149.

Whitmore, Herndon, *J. Am. Chem. Soc.*, 1933, **55**, 3430.

Klepper, *Chimie et Industrie*, 1929, Special No., 261.

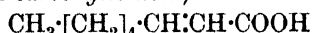
Maman, *Chem. Zentr.*, 1936, I, 2332.

See also last reference above.

**1-Octene-1-carboxylic Acid.**

See 1-Nonenic Acid.

**1-Octenic Acid** (2-n-Amylacrylic acid, 1-heptene-1-carboxylic acid)



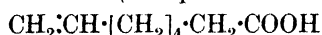
$\text{C}_8\text{H}_{14}\text{O}_2$  MW, 142

Nitrile:  $\text{C}_8\text{H}_{13}\text{N}$ . MW, 123. (a) *Cis*: b.p. 74-5°/11 mm.  $D_4^{20}$  0.82854.  $n_D^{20}$  1.44032. (b) *Trans*: m.p. 57.6-58°. B.p. 84°/11 mm.  $D_4^{20}$  0.82815.  $n_D^{20}$  1.44344.

Amide:  $\text{C}_8\text{H}_{15}\text{ON}$ . MW, 141. (a) *Cis*: m.p. 66.0-66.4°. (b) *Trans*: m.p. 134.7-135.1°.

Bruylants, Fonteyn, *Chem. Abstracts*, 1933, 27, 5717.

**6-Octenic Acid** (1-Heptene-7-carboxylic acid)

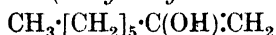


$\text{C}_8\text{H}_{14}\text{O}_2$  MW, 142

Et ester:  $\text{C}_{10}\text{H}_{18}\text{O}_2$ . MW, 170. B.p. 210°.

Carmichael, *J. Chem. Soc.*, 1922, 121, 2549.

**1-Octenol-2** (2-Hydroxy-1-octene)

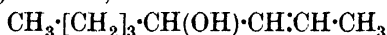


$\text{C}_8\text{H}_{16}\text{O}$  MW, 128

Me ether:  $\text{C}_9\text{H}_{18}\text{O}$ . MW, 142. B.p. 166-8°.  $D_4^{16}$  0.8170.  $n_D^{16}$  1.4309.

Moureu, *Compt. rend.*, 1904, 138, 287.

**2-Octenol-4** (4-Hydroxy-2-octene, butyl-propenylcarbinol)

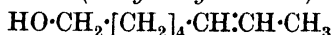


$\text{C}_8\text{H}_{16}\text{O}$  MW, 128

B.p. 93.9-95.9°/40 mm.

Mulliken, Wakeman, Gerry, *J. Am. Chem. Soc.*, 1935, 57, 1606.

**2-Octenol-8** (8-Hydroxy-2-octene)

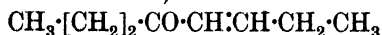


$\text{C}_8\text{H}_{16}\text{O}$  MW, 128

B.p. 187-93°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

Löbl, *Monatsh.*, 1903, 24, 398.

**3-Octenone-5** (Propyldenemethyl n-propyl ketone, 5-keto-3-octene)

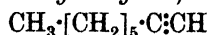


$\text{C}_8\text{H}_{14}\text{O}$  MW, 126

B.p. 68-72°/25 mm.

Benary, *Ber.*, 1931, 64, 2544.

**1-Octine** (n-Hexylacetylene)

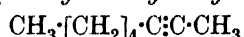


$\text{C}_8\text{H}_{14}$  MW, 110

B.p. 127.6-128°.

Mulliken, Wakeman, Gerry, *J. Am. Chem. Soc.*, 1935, 57, 1607.

**2-Octine** (Methyl-n-amylacetylene)

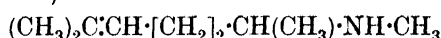


$\text{C}_8\text{H}_{14}$  MW, 110

B.p. 138-138.4°.

See previous reference.

**Octinum** (Octin, 6-methylamino-2-methyl-heptene-2)



$\text{C}_9\text{H}_{19}\text{N}$  MW, 141

B.p. 176-8°. Soporific.

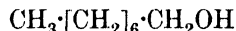
Picrate: m.p. 70°.

Knoll, Danish P., 48,717, (*Chem. Zentr.*, 1934, II, 1493).

**n-Octoic Acid.**

See n-Caprylic Acid.

**n-Octyl Alcohol** (1-Octanol, 1-hydroxy-n-octane)



$\text{C}_8\text{H}_{18}\text{O}$  MW, 130

M.p. -16.7°. B.p. 194.45° (195-8°, 195-7°), 98°/19 mm., 90.2°/11.8 mm., 82.8°/7.5 mm.  $D_4^{20}$  0.8270.  $n_D^{20}$  1.43035. Heat of comb.  $\text{C}_p$  1265.0 Cal.,  $\text{C}_p$  1262.7 Cal.

3:5-Dinitrobenzoyl: m.p. 61-2°.

Me ether: see Methyl octyl Ether.

Et ether:  $\text{C}_{10}\text{H}_{22}\text{O}$ . MW, 158. B.p. 189.2°, 72-3°/8 mm.

n-Propyl ether:  $\text{C}_{11}\text{H}_{24}\text{O}$ . MW, 172. B.p. 207°.

n-Butyl ether:  $\text{C}_{12}\text{H}_{26}\text{O}$ . MW, 186. B.p. 225-7°.

n-Heptyl ether:  $\text{C}_{15}\text{H}_{32}\text{O}$ . MW, 228. B.p. 278.8°.

Phenylurethane: m.p. 74-74.5° (72°).

o-Nitrophenylurethane: m.p. 44°.

m-Nitrophenylurethane: m.p. 63°.

3:5-Dinitrophenylurethane: m.p. 69°.

Blaise, Picard, *Compt. rend.*, 1911, 152, 269.

Reichstein, Ammann, Trivelli, *Helv. Chim. Acta*, 1932, 15, 267.

Schrauth, Schenck, Stickdorn, *Ber.*, 1931, 64, 1318.

Goebel, Marvel, *J. Am. Chem. Soc.*, 1933, 55, 1696.

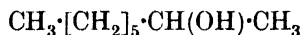
Vaughn, Spahr, Nieuwland, *J. Am. Chem. Soc.*, 1933, 55, 4207.

Deutsche Gold- u. Silber-Scheideanstalt, E.P., 381,185, (*Chem. Abstracts*, 1933, 27, 3943).

Ruhoff, Reid, *J. Am. Chem. Soc.*, 1933, 55, 3825.

Deffert, *Bull. soc. chim. Belg.*, 1931, 40, 385.

**sec.-n-Octyl Alcohol** (*Methyl-n-hexylcarbinol*, *2-hydroxy-n-octane*, *octanol-2*, *capryl alcohol*)



$\text{C}_8\text{H}_{18}\text{O}$  MW, 130

d.-

B.p. 86°/20 mm.  $[\alpha]_D^{17} + 9.9^\circ$ .  $D_4^{20}$  0.8216.

l.-

B.p. 86°/20 mm.  $[\alpha]_D^{17} - 9.9^\circ$ .

dl.-

B.p. 179.5°, 110°/120 mm.  $D_4^{25.2}$  0.8188.  $n_D^{20}$  1.42025.

*Formyl*:  $\text{C}_9\text{H}_{18}\text{O}_2$ . MW, 158. B.p. 184°/744 mm.  $D_4^{14}$  0.8642.

*Acetyl*:  $\text{C}_{10}\text{H}_{20}\text{O}_2$ . MW, 172. B.p. 194.5°/744 mm., 84°/15 mm.  $D_4^{19}$  0.8606.  $n_D^{20}$  1.4141.

*Bromoacetyl*:  $\text{C}_{10}\text{H}_{19}\text{O}_2\text{Br}$ . MW, 251. B.p. 137°/18 mm.

*Propionyl*:  $\text{C}_{11}\text{H}_{22}\text{O}_2$ . MW, 186. B.p. 211°/744 mm., 96°/15 mm.  $D_{44}^{20.5}$  0.8650.  $n_D^{20}$  1.4168.

*Butyryl*:  $\text{C}_{12}\text{H}_{24}\text{O}_2$ . MW, 200. B.p. 220°/744 mm., 115°/18 mm.  $D_4^{17}$  0.8587.  $n_D^{20}$  1.4202.

*Isobutyryl*: b.p. 220°/744 mm.  $D_4^{14}$  0.8554.

*Valeryl*:  $\text{C}_{13}\text{H}_{26}\text{O}_2$ . MW, 214. B.p. 127°/17 mm.  $D_4^{16.5}$  0.8560.  $n_D^{20}$  1.4225.

*Isovaleryl*: b.p. 236.5°/744 mm.  $D_4^{14}$  0.8540.

*d-Caproyl*:  $\text{C}_{14}\text{H}_{28}\text{O}_2$ . MW, 228. B.p. 141°/17 mm.  $D_4^{12}$  0.8598.  $n_D^{20}$  1.4264.

*d-Heptyl*:  $\text{C}_{15}\text{H}_{30}\text{O}_2$ . MW, 242. B.p. 151°/15 mm.  $D_4^{13}$  0.8593.  $n_D^{20}$  1.4290.

*d-Capryl*:  $\text{C}_{16}\text{H}_{32}\text{O}_2$ . MW, 256. B.p. 165°/18 mm.  $D_4^{12}$  0.8557.  $n_D^{20}$  1.4308.

*d-Nonoyl*:  $\text{C}_{17}\text{H}_{34}\text{O}_2$ . MW, 270. B.p. 174°/17 mm.  $D_4^{22}$  0.8542.  $n_D^{20}$  1.4342.

*d-Undecyl*:  $\text{C}_{19}\text{H}_{38}\text{O}_2$ . MW, 298. B.p. 167°/3 mm.  $D_4^{19}$  0.8538.  $n_D^{20}$  1.4373.

*d-Lauryl*:  $\text{C}_{20}\text{H}_{40}\text{O}_2$ . MW, 312. B.p. 183°/7 mm.  $D_4^{16.5}$  0.8585.  $n_D^{20}$  1.4393.

*d-Myristyl*:  $\text{C}_{22}\text{H}_{44}\text{O}_2$ . MW, 340. B.p. 197°/4 mm.  $D_4^{17}$  0.8562.  $n_D^{20}$  1.4416.

*d-Palmityl*:  $\text{C}_{24}\text{H}_{48}\text{O}_2$ . MW, 368. M.p. 32°. B.p. 216°/18 mm.  $D_4^{16}$  0.8372.  $n_D^{20}$  1.4438.

*d-Stearyl*:  $\text{C}_{26}\text{H}_{52}\text{O}_2$ . MW, 396. M.p. 34°. B.p. 235°/6 mm.  $D_4^{18}$  0.8410.

*Phenylacetyl*:  $\text{C}_{16}\text{H}_{24}\text{O}_2$ . MW, 248. B.p. 195°/35 mm., 174°/12 mm.  $D_4^{20}$  0.9578.

*Benzoyl*:  $\text{C}_{15}\text{H}_{20}\text{O}_2$ . MW, 234. B.p. 175°/2-5 mm.  $D_4^{20}$  1.0185.  $n_D^{20}$  1.5476.

*l-o-Aminobenzoyl*:  $\text{C}_{15}\text{H}_{23}\text{O}_2\text{N}$ . MW, 249. B.p. 183°/10 mm.

*l-m-Aminobenzoyl*: b.p. 195-9°/18 mm.

*l-p-Aminobenzoyl*: m.p. 69-70°. B.p. 200°/10 mm. *B,HCl*: m.p. 131-3°.

*Salicyloyl*:  $\text{C}_{15}\text{H}_{22}\text{O}_3$ . MW, 250. B.p. 170°/10 mm.

*Me ether*:  $\text{C}_9\text{H}_{20}\text{O}$ . MW, 144. B.p. 76-7°/44 mm.  $D_4^{20}$  0.8094.  $n_D^{25}$  1.4212.

*Et ether*:  $\text{C}_{10}\text{H}_{22}\text{O}$ . MW, 158. B.p. 63-5°/14 mm.  $D_4^{20}$  0.7861.  $n_D^{25}$  1.4136.

*n-Propyl ether*:  $\text{C}_{11}\text{H}_{24}\text{O}$ . MW, 172. B.p. 76°/18 mm.  $D_4^{20}$  0.7887.  $n_D^{25}$  1.4148.

*n-Butyl ether*:  $\text{C}_{12}\text{H}_{26}\text{O}$ . MW, 186. B.p. 85-6°/14 mm.  $D_4^{20}$  0.7923.  $n_D^{25}$  1.4168.

*n-Amyl ether*:  $\text{C}_{13}\text{H}_{28}\text{O}$ . MW, 200. B.p. 99°/15 mm.  $D_4^{20}$  0.7958.  $n_D^{25}$  1.4218.

*n-Hexyl ether*:  $\text{C}_{14}\text{H}_{30}\text{O}$ . MW, 214. B.p. 115°/15 mm.  $D_4^{20}$  0.7983.  $n_D^{25}$  1.4252.

*n-Heptyl ether*:  $\text{C}_{15}\text{H}_{32}\text{O}$ . MW, 228. B.p. 129°/18 mm.  $D_4^{20}$  0.8017.  $n_D^{25}$  1.4267.

*n-Octyl ether*:  $\text{C}_{16}\text{H}_{34}\text{O}$ . MW, 242. B.p. 146°/13 mm.  $D_4^{20}$  0.8038.  $n_D^{25}$  1.4301.

*n-Nonyl ether*:  $\text{C}_{17}\text{H}_{36}\text{O}$ . MW, 256. B.p. 163°/18 mm.  $D_4^{20}$  0.8042.  $n_D^{25}$  1.4325.

*Benzyl ether*:  $\text{C}_{15}\text{H}_{24}\text{O}$ . MW, 220. B.p. 154°/18 mm.  $D_4^{20}$  0.8974.

*Diphenylmethyl ether*:  $\text{C}_{21}\text{H}_{28}\text{O}$ . MW, 296. B.p. 202°/10 mm.  $D_4^{17}$  0.9675.

*Triphenylmethyl ether*:  $\text{C}_{27}\text{H}_{32}\text{O}$ . MW, 372. B.p. 178°/0.3 mm.  $D_4^{16}$  1.026.

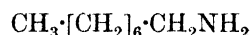
Kenyon, McNicol, *J. Chem. Soc.*, 1923, 123, 18.

Kenyon, *Organic Syntheses*, 1926, VI, 68.

Waterman, te Nuyl, *Rec. trav. chim.*, 1932, 51, 533.

Ellis, Reid, *J. Am. Chem. Soc.*, 1932, 54, 1678.

#### n-Octylamine (l-Amino-n-octane)



$\text{C}_8\text{H}_{19}\text{N}$  MW, 129

B.p. 175-7°/745 mm. (179-80°, 185-7°).  $D_4^{20.5}$  0.7769.

*Picrate*: m.p. 111.5-112.5°.

Nyssens, *Chem. Abstracts*, 1931, 25, 70.

Adamson, Kenner, *J. Chem. Soc.*, 1934, 842.

#### sec.-n-Octylamine (2-Amino-n-octane)



$\text{C}_8\text{H}_{19}\text{N}$  MW, 129

B.p. 172-5° (164-5°).  $D_4^{20}$  0.7745.  $n_D^{25}$  1.42319.

Kishner, *Chem. Zentr.*, 1900, I, 653.

#### n-Octylbenzene (l-Phenyl-n-octane)



$\text{C}_{14}\text{H}_{22}$  MW, 190

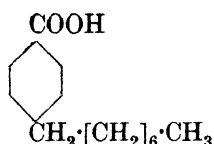
F.p. — 7°. B.p. 264–5°, (262–4°, 261–3°, 257°).  $D_4^{20}$  0.8583.  $\text{CrO}_3 \longrightarrow$  benzoic acid.

Sabatier, Maible, *Compt. rend.*, 1914, **158**, 834.

Tilicheev, Kuruindin, *Chem. Abstracts*, 1931, **25**, 3469.

Eisenlohr, Schultz, *Ber.*, 1924, **57**, 1815.

**p-Octylbenzoic Acid**

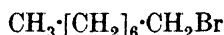


$\text{C}_{15}\text{H}_{22}\text{O}_2$  MW, 234

Leaflets from hot EtOH. M.p. 139°. Spar. sol.  $\text{H}_2\text{O}$ .

Beran, *Ber.*, 1885, **18**, 138.

**n-Octyl bromide (1-Bromo-n-octane)**



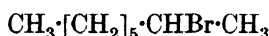
$\text{C}_8\text{H}_{17}\text{Br}$  MW, 193

M.p. — 55.0°. B.p. 201.5°.  $D_4^{25}$  1.10788.  $n_D^{25}$  1.4503.

Kamm, Marvel, *J. Am. Chem. Soc.*, 1920, **42**, 309.

*Organic Syntheses*, 1921, I, 7; Collective Vol. I, 28.

**sec.-n-Octyl bromide (2-Bromo-n-Octane)**



$\text{C}_8\text{H}_{17}\text{Br}$  MW, 193

d-.  
B.p. 71°/14 mm., 60°/3 mm.  $D_4^{25}$  1.0982.  $n_D^{25}$  1.4475.  $[\alpha]_D^{25} + 34.2^\circ$  (+ 29.8°).

l-.  
B.p. 83–4°/18 mm.  $n_D^{20}$  1.4500.  $[\alpha]_D^{25} - 33.1^\circ$  (– 34.25°).

dl-.  
B.p. 72°/14 mm., 61°/3 mm.  $D_4^{25}$  1.0878 (1.09681).  $n_D^{25}$  1.4442 (1.4482).

Ellis, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 1680.

Rulé, Smith, Harrower, *J. Chem. Soc.*, 1933, 386.

Shriner, Young, *J. Am. Chem. Soc.*, 1930, **52**, 3332.

Reynolds, Adkins, *J. Am. Chem. Soc.*, 1929, **51**, 279.

Tseng, Hau, Hu, *Chem. Zentr.*, 1936, I, 2917.

**n-Octyl chloride (1-Chloro-n-octane)**



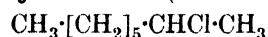
$\text{C}_8\text{H}_{17}\text{Cl}$  MW, 148.5

B.p. 184°, 78°/15 mm.  $D_4^0$  0.892.

Bouveault, Blanc, *Bull. soc. chim.*, 1904, **31**, 673.

Clark, Streight, *Chem. Abstracts*, 1930, **24**, 586.

**sec.-n-Octyl chloride (2-Chloro-n-octane)**



$\text{C}_8\text{H}_{17}\text{Cl}$  MW, 148.5

d-.  
B.p. 75°/28 mm., 55–6°/10–11 mm.  $D_4^{17}$  0.8658.  $n_D^{21}$  1.4273.  $[\alpha]_D^{17} + 20.40^\circ$  ( $[\alpha]_D^{20} + 33.70^\circ$ ).

l-.  
B.p. 70°/25 mm., 68°/21 mm., 60°/12 mm.  $D_4^{17}$  0.8628.  $n_D^{20}$  1.4302.  $[\alpha]_D^{17} - 20.44^\circ$ .

dl-.  
B.p. 171–3°.  $D^{15}$  0.87075.

Houssa, Phillips, *J. Chem. Soc.*, 1932, 109.  
Malbot, *Bull. soc. chim.*, 1890, **3**, 69.

Houssa, Kenyon, Phillips, *J. Chem. Soc.*, 1929, 1700.

Rule, Smith, Harrower, *J. Chem. Soc.*, 1933, 386.

See also last reference above.

**Octylene.**

See Octene.

**Octyl hexadecyl Ketone (Pentacosanone-9)**

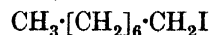


$\text{C}_{25}\text{H}_{50}\text{O}$  MW, 366

Powder from  $\text{C}_6\text{H}_6$ . M.p. 66°. Sol. EtOH.  
*Semicarbazone*: m.p. 39–41°.

Brigl, *Z. physiol. Chem.*, 1915, **95**, 178.

**n-Octyl iodide (1-Iodo-n-octane)**

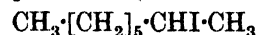


$\text{C}_8\text{H}_{17}\text{I}$  MW, 240

M.p. — 45.7°. B.p. 225–225.5°, 194°/330 mm.  $D_4^{15}$  1.33568.

Möslinger, *Ann.*, 1877, **185**, 55.

**sec.-n-Octyl iodide (2-Iodo-n-octane)**



$\text{C}_8\text{H}_{17}\text{I}$  MW, 240

d-.  
B.p. 101°/22 mm.  $D_4^{17}$  1.3314.  $[\alpha]_D^{17} + 39.83^\circ$ .

l-.  
B.p. 92°/12 mm.  $D_4^{17}$  1.3299.  $[\alpha]_D^{17} - 40.56^\circ$ .

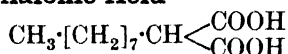
dl-.

B.p. 190° (210°) part. decomp.  $D_{15}^{25}$  1.318.

Pickard, Kenyon, *J. Chem. Soc.*, 1911, 99, 69.

Cf. Houssa, Kenyon, Phillips, *J. Chem. Soc.*, 1929, 1700.

**n-Octylmalonic Acid**



$\text{C}_{11}\text{H}_{20}\text{O}_4$

MW, 216

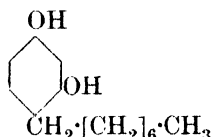
Prisms from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 115° (108°).

Di-Et ester:  $\text{C}_{15}\text{H}_{28}\text{O}_4$ . MW, 272. B.p. 169°/17 mm.

Robinson, *J. Chem. Soc.*, 1924, 125, 228.

Clutterbuck, Raistrick, Rintoul, *Trans. Roy. Soc.*, 1931, B, 220, 301.

**4-n-Octylresorcinol** (2 : 4-Dihydroxyoctylbenzene)



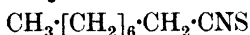
$\text{C}_{14}\text{H}_{22}\text{O}_2$

MW, 222

M.p. 74-5°. B.p. 199-201°/6-7 mm.

Dohme, Cox, Miller, *J. Am. Chem. Soc.*, 1926, 48, 1692.

**n-Octyl thiocyanate**



$\text{C}_9\text{H}_{17}\text{NS}$

MW, 171

B.p. 141-2°/19 mm.  $D_4^{25}$  0.9149.  $n_D^{25}$  1.4642.

Allen, *J. Am. Chem. Soc.*, 1935, 57, 198.

**1-Octyl-2-undecyl-ethylene.**

See 9-Heneicosene.

**Œnanthaldehyde.**

See n-Heptaldehyde.

**Œnanthic Acid.**

See n-Heptylic Acid.

**Œnanthol.**

See n-Heptaldehyde.

**Œnanthone.**

See Di-n-hexyl Ketone.

**Œnanthylic Acid.**

See n-Heptylic Acid.

**Œnanthylidene** (Œnanthine, n-amylacetylene, 1-heptine)



$\text{C}_7\text{H}_{12}$

MW, 96

F.p. below -70°. B.p. 99-100° (108-10°), 26°/10 mm.  $D^{19}$  0.750.  $n_D^{19}$  1.418. Red. →

n-heptane.  $\text{NH}_3 \cdot \text{AgNO}_3 \rightarrow$  white ppt.  
 $\text{NH}_3 \cdot \text{Cu}_2\text{Cl}_2 \rightarrow$  yellow ppt.

Moureu, André, *Ann. chim.*, 1914, 1, 116 (Footnote).

Bourguel, *Ann. chim.*, 1925, 3, 191, 325.

Bouis, *Ann. chim.*, 1928, 9, 461.

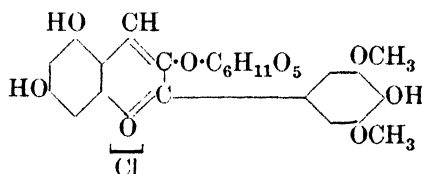
Hill, Tyson, *J. Am. Chem. Soc.*, 1928, 50, 172.

Chem. Fabrik Flörsheim, *Chem. Abstracts*, 1912, 6, 2072.

**Œnidin chloride.**

Malvidin chloride, q.v.

**Œnin chloride** (Primulin chloride, malvidin chloride 3-β-glucoside)



$\text{C}_{23}\text{H}_{25}\text{O}_{12}\text{Cl}$

MW, 528.5

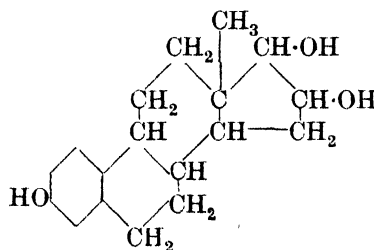
Pigment of skins of black grapes. Violet cryst. by transmitted light with yellow metallic cast. Bronze appearance in masses. Gives bluish-violet smear on paper or porcelain.

Picrate: red needles with green reflex. Decomp. at 202°.

Kondo, *Chem. Abstracts*, 1930, 24, 2748.

Levy, Posternack, Robinson, *J. Chem. Soc.*, 1931, 2701 (Bibl.).

**Œstriol** (Theelol, follicular hormone hydrate)



$\text{C}_{18}\text{H}_{24}\text{O}_3$

MW, 288

Isolated from urine of pregnancy. Cryst. from EtOH-AcOEt. M.p. 283° (275°). Sol. 33,000 parts  $\text{H}_2\text{O}$ . Sol. EtOH,  $\text{Me}_2\text{CO}$ , Py. Mod. sol.  $\text{Et}_2\text{O}$ . Spar. sol. pet. ether. Insol. alk. carbonates. Very sol. aq. alkalis.  $[\alpha]_{D}^{25} + 71^\circ$  in EtOH,  $[\alpha]_D + 34.4^\circ$  in Py. Heat in vacuo with  $\text{KHSO}_4 \rightarrow$  œstrone. Forms Me ether with diazomethane.

Me ether: cryst. from EtOH. M.p. 162.5-164°.



*Triacetyl*: m.p. 127°. Ten times as physiologically active as oestriol.

Cartland, Meyer, Miller, Rutz, *J. Biol. Chem.*, 1935, **109**, 215.

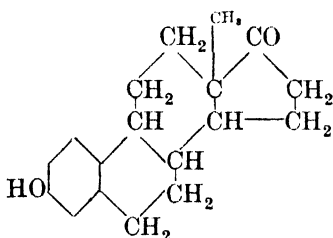
MacCorquodale, Thayer, Doisy, *J. Biol. Chem.*, 1933, **99**, 329.

Marrian, Haslewood, *Biochem. J.*, 1932, **26**, 25.

Butenandt, Hildebrandt, *Z. physiol. Chem.*, 1931, **199**, 245.

Marrian, *Biochem. J.*, 1930, **24**, 435, 1021.

**Estrone** (*Estrin, theelin, ketohydroxyaestrin, menformon, folliculin, follicular hormone, α-follicular hormone, progynon*)



$C_{18}H_{22}O_2$

MW, 270

The oestrus-producing hormone obtained, among other sources, from urine of pregnancy. Colourless cryst. from EtOH. Three polymorphic forms, m.ps. 254°, 256°, 259°. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. Et<sub>2</sub>O, AcOEt. Spar. sol. pet. ether. Insol. H<sub>2</sub>O. Sol. alkalis. Insol. alk. carbonates.  $[\alpha]_D^{25} + 158.5^\circ$ . Distils undecomp. at 150–200°/0.002 mm. Does not give Liebermann–Burchard reaction. Gives no col. with FeCl<sub>3</sub>.

*Me ether*: C<sub>19</sub>H<sub>24</sub>O<sub>2</sub>. MW, 284. Cryst. from EtOH. M.p. 167.5–169.5°. Physiologically inactive. *Semicarbazone*: cryst. from EtOH. M.p. 267° decomp.

*Acetyl*: needles or plates from EtOH.Aq. M.p. 126°. Physiologically active.

*Benzoyl*: needles from MeOH. M.p. 217.5°. Physiologically active.

*Oxime*: C<sub>18</sub>H<sub>23</sub>O<sub>2</sub>N. MW, 285. Needles from EtOH.Aq. Decomp. at 233°. Physiologically active.

*Semicarbazone*: cryst. from EtOH. M.p. 258–60° decomp. Physiologically inactive.

Marker, Kamm, Oakwood, Laucius, *J. Am. Chem. Soc.*, 1936, **58**, 1503.

Cartland, Meyer, Miller, Rutz, *J. Biol. Chem.*, 1935, **109**, 213.

Curtis, MacCorquodale, Thayer, Doisy, *J. Biol. Chem.*, 1934, **107**, 191.

Kofler, Hauschild, *Z. physiol. Chem.* 1934, **224**, 150.

Butenandt, Störmer, *Z. physiol. Chem.*, 1932, **208**, 129, 149.

Doisy, Veler, Thayer, *J. Biol. Chem.*, 1930, **86**, 499; **87**, 357.

Butenandt, Ziegner, *Z. physiol. Chem.*, 1930, **188**, 1.

**Oleandrin**

C<sub>31</sub>H<sub>48</sub>O<sub>9</sub>

MW, 564

Glucoside from leaves of *Nerium oleander* (Laurier-rose), Linn. Cryst. from hot MeOH.Aq. M.p. 70–5°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Sublimes.

Windaus, Westphal, *Chem. Abstracts*, 1927, **21**, 299.

Tanret, *Compt. rend.*, 1932, **194**, 914.

Kahlbaum, D.R.P., 577,257, (*Chem. Abstracts*, 1933, **27**, 4032).

**Oleanic Acid.**

See Oleanolic Acid.

**Oleanol**

C<sub>29</sub>H<sub>48</sub>O

MW, 412

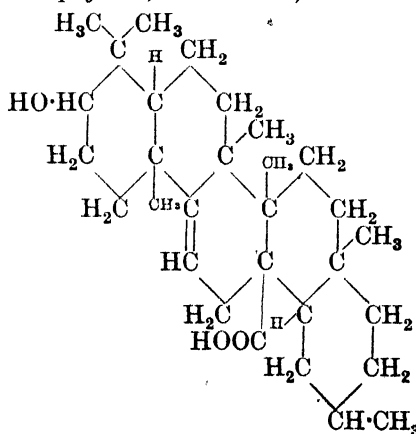
Needles from Me<sub>2</sub>CO. M.p. 216–20°.  $[\alpha]_D^{20} + 59^\circ$  in CHCl<sub>3</sub>. Gives Liebermann reaction.

*Acetyl*: needles from EtOH. M.p. 209–10°.  $[\alpha]_D^{21} + 44.7^\circ$  in CHCl<sub>3</sub>.

Winterstein, Stein, *Z. physiol. Chem.*, 1931, **202**, 222.

Kuwada, *Chem. Abstracts*, 1936, **28**, 8237.

**Oleanolic Acid** (*Panax sapogenin, guagenin, sugar beet sapogenin, "oleanol," caryophyllin, mistletoe sapogenin, oleanic acid*)



Suggested structure

C<sub>30</sub>H<sub>48</sub>O<sub>3</sub>

MW, 456

Occurs as glucoside in leaves of *Aralia japonica*, (Thunb.), mistletoe, cloves, sugar beet,

olive leaves, etc. Prisms from EtOH. M.p. 306–8° (305°). Sol. MeOH, EtOH.  $[\alpha]_D^{25} + 79.5^\circ$  in  $\text{CHCl}_3$ .

*Acetyl*: m.p. 268° (258–60°).

*Diphenylurethane*: m.p. 137–8°.

*Me ester*:  $\text{C}_{31}\text{H}_{50}\text{O}_2$ . MW, 470. M.p. 196–8° (184°). *Acetyl*: m.p. 223°. *Oxime*: m.p. 246° decomp. (226°).

Kitasato, *Chem. Abstracts*, 1934, **28**, 4051.

Aumüller, Schicke, Wedekind, *Ann.*, 1935, **517**, 211.

Ruzicka, Hofmann, *Helv. Chim. Acta*, 1936, **19**, 114.

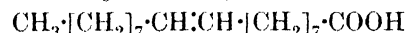
Jacobs, Fleck, *J. Biol. Chem.*, 1932, **96**, 341.

Van der Haar, *Rec. trav. chim.*, 1927, **46**, 775, 793.

Ruzicka, Goldberg, Hofmann, *Helv. Chim. Acta*, 1937, **20**, 325.

*Note*.—The "Oleanol" described by Power, Tutin, *J. Chem. Soc.*, 1908, **93**, 896, appears to be identical with oleanolic acid.

**Oleic Acid** (*Cis*-8-*Heptadecylenecarboxylic acid*,  $\Delta^9$ -octadecylenic acid)



$\text{C}_{18}\text{H}_{34}\text{O}_2$  MW, 282

F.p. 11.8–12.2° (13.2°). *Labile form*: m.p. 12°. *Stable form*: m.p. 16° (15.4°). B.p. 285.5–286°/100 mm., 203–5°/5 mm., 170–5°/2–3 mm. Sol. EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .  $D^{20}_D$  0.898.  $n^{20}_D$  1.45823 (1.4610). Heat of comb.  $\text{C}_p$  2682.0 Cal.,  $\text{C}_v$  2677.6 Cal.

*Me ester*:  $\text{C}_{19}\text{H}_{36}\text{O}_2$ . MW, 296. B.p. 212–13°/15 mm., 160–2°/2–3 mm.  $D^{18}$  0.879.

*Et ester*:  $\text{C}_{20}\text{H}_{38}\text{O}_2$ . MW, 310. B.p. 216–17°/151 mm.  $D^{25}$  0.8671.

*Propyl ester*:  $\text{C}_{21}\text{H}_{40}\text{O}_2$ . MW, 324. B.p. 266–7°/15 mm.

*Isopropyl ester*: b.p. 223–4°/15 mm.

*Butyl ester*:  $\text{C}_{22}\text{H}_{42}\text{O}_2$ . MW, 338. B.p. 227–8°/15 mm. (235–40°/10–18 mm.).

*Isobutyl ester*: *tebelon*. B.p. 226–7°/10 mm., 190°/4 mm.  $D^{20}$  0.86.

*tert.-Butyl ester*: b.p. 223–224.5°/10 mm.

*Isoamyl ester*:  $\text{C}_{23}\text{H}_{44}\text{O}_2$ . MW, 352. B.p. 223–4°/10 mm.  $D^{16}$  0.897.

*tert.-Amyl ester*: b.p. 223–225.5°/10 mm.

$\alpha$ -*Glyceryl ester*: *see*  $\alpha$ -Mono-olein.

*Vinyl ester*:  $\text{C}_{20}\text{H}_{36}\text{O}_2$ . MW, 308. B.p. 173°/2 mm.

*Allyl ester*:  $\text{C}_{21}\text{H}_{38}\text{O}_2$ . MW, 322. B.p. 219–21°/10 mm.

*Phenyl ester*:  $\text{C}_{24}\text{H}_{38}\text{O}_2$ . MW, 358. B.p. about 230°/7 mm.

*m-Tolyl ester*:  $\text{C}_{25}\text{H}_{40}\text{O}_2$ . MW, 372. B.p. about 240°/5 mm.

*p-Chlorophenacyl ester*:  $\text{C}_{26}\text{H}_{40}\text{O}_2\text{Cl}$ . MW, 419.5. M.p. 40°.

*p-Bromophenacyl ester*:  $\text{C}_{26}\text{H}_{40}\text{O}_2\text{Br}$ . MW, 464. M.p. 40°.

*p-Phenylphenacyl ester*:  $\text{C}_{32}\text{H}_{45}\text{O}_2$ . MW, 461. M.p. 61°.

*Menthyl ester*:  $\text{C}_{28}\text{H}_{52}\text{O}_2$ . MW, 420. B.p. about 240°/4 mm.

*Stigmasteryl ester*:  $\text{C}_{48}\text{H}_{82}\text{O}_2$ . MW, 690. M.p. 44°.

*Chloride*:  $\text{C}_{18}\text{H}_{33}\text{OCl}$ . MW, 300.5. B.p. 213°/13–15 mm., 200°/11 mm., 190°/9 mm.

*Amide*:  $\text{C}_{18}\text{H}_{35}\text{ON}$ . MW, 281. M.p. 75–6°.

*Anhydride*:  $\text{C}_{36}\text{H}_{66}\text{O}_3$ . MW, 546. M.p. 22–4°.  $D^{18}$  0.900.  $n^{20}_D$  1.4630.

*Nitrile*:  $\text{C}_{18}\text{H}_{33}\text{N}$ . MW, 263. B.p. 330–5° decomp.

*Anilide*: m.p. 41°. B.p. 143.5°/10 mm.

*Phenylhydrazide*: m.p. 72–3°.

Noller, Bannerot, *J. Am. Chem. Soc.*, 1934, **56**, 1563.

Koyama, *Chem. Abstracts*, 1932, **26**, 5067.

Bannister, U.S.P., 1,796,231, (*Chem. Abstracts*, 1931, **25**, 2441).

Skillon, *J. Soc. Chem. Ind.*, 1931, **50**, 131t.

Raymond, *Chimie et Industrie*, Special No., 1929, 523.

Scheffers, *Rec. trav. chim.*, 1927, **46**, 293.

Bertram, *ibid.*, 397.

Holde, Gorgas, *Z. angew. Chem.*, 1926, **39**, 1443.

Robinson, Robinson, *J. Chem. Soc.*, 1925, 175.

Holde, Rietz, *Ber.*, 1924, **57**, 99.

Maihle, *Bull. soc. chim.*, 1920, **27**, 226.

Preiswerk, U.S.P., 1,318,461, (*Chem. Abstracts*, 1920, **14**, 95).

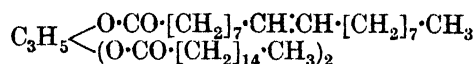
Täufel, Kunkle, *Chem. Zentr.*, 1935, **I**, 2971.

Brown, Shinowara, *J. Am. Chem. Soc.*, 1937, **59**, 6.

### Oleic Alcohol.

*See* Octadecenyl Alcohol.

### Oleodipalmitin



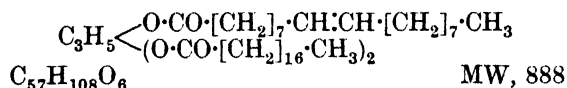
$\text{C}_{53}\text{H}_{100}\text{O}_6$  MW, 832

Occurs in soya bean oil, butter fat, cocoa fat, etc.

(i) M.p. 27–8°. (ii) Cryst. from  $\text{Me}-\text{C}_2\text{O}$

CHCl<sub>3</sub>. M.p. 38° (37°). Remelts at 28°.  
(iii) M.p. 48°.

Hashi, *Chem. Zentr.*, 1928, I, 1470.  
Amberger, *Chem. Abstracts*, 1919, 13, 3252.

**Oleodistearin**

Occurs in fat of seeds of *Mangifera indica*, Linn. M.p. 44° (42°, 29–31°).

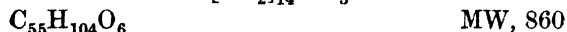
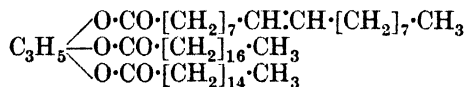
Amberger, Bromig, *Biochem. Z.*, 1922, 130, 252.

**Oléone** (*Diheptadecenyl ketone*)

(i) Pale yellow cryst. from EtOH. M.p. 58.5°. (ii) Pale yellow oil. B.p. 120–75°/20 mm. Red. → pentatriacontane.

Oxime : m.p. 31°.

Breuer, Weinmann, *Monatsh.*, 1935, 67, 42.

**Oleopalmitostearin**

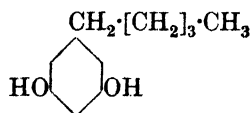
(i) M.p. 31.3°. (ii) M.p. 42°.

Klimont, *Monatsh.*, 1902, 23, 55.

Hansen, *Chem. Zentr.*, 1902, I, 1116.

**Oleyl Alcohol.**

See Octadecenyl Alcohol.

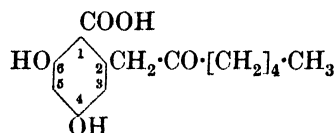
**Olivetol** (5-*n*-Amylresorcinol)

Prisms + 1H<sub>2</sub>O. M.p. 40–1°. Sol. ord. org. solvents.

*Di-Me ether* :  $\text{C}_{13}\text{H}_{20}\text{O}_2$ . MW, 208. B.p. 114°/2 mm.

Asahina, Asano, *Ber.*, 1932, 65, 478.

Asahina, Nogami, *Ber.*, 1935, 68, 1501.

**Olivetonic Acid**

Needles from hot H<sub>2</sub>O. M.p. 159–60°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, hot H<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin.

*Me ester* :  $\text{C}_{15}\text{H}_{20}\text{O}_5$ . MW, 280. M.p. 85–6°.

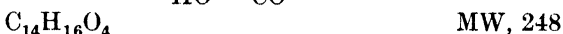
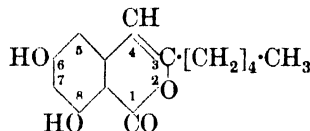
*4-Me ether* :  $\text{C}_{15}\text{H}_{20}\text{O}_5$ . MW, 280. M.p. 94–5°.

*Me ester* :  $\text{C}_{16}\text{H}_{22}\text{O}_5$ . MW, 294. M.p. 80°.

*6-Me ether* : m.p. 119–20°.

*Di-Me ether* :  $\text{C}_{16}\text{H}_{22}\text{O}_5$ . MW, 294. Needles from EtOH.Aq. M.p. 93°. *Oxime* : m.p. 120°.

Asahina, Fuzikawa, *Ber.*, 1935, 68, 2023.

**Olivetonicide**

Needles from ligroin. M.p. 110°.

*6-Me ether* :  $\text{C}_{15}\text{H}_{18}\text{O}_4$ . MW, 262. Prisms from EtOH. M.p. 57°. *Acetyl* : m.p. 60–1°.

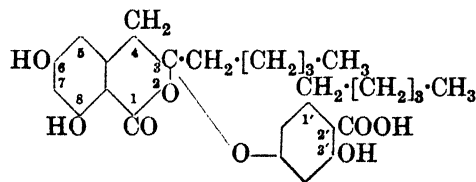
*8-Me ether* : needles from C<sub>6</sub>H<sub>6</sub>. M.p. 146–7°. *Acetyl* : m.p. 84°.

*Di-Me ether* :  $\text{C}_{16}\text{H}_{20}\text{O}_4$ . MW, 276. Prisms from EtOH. M.p. 94°.

*6-Acetyl* : m.p. 55°.

*Diacetyl* : m.p. 59°.

Asahina, Fuzikawa, *Ber.*, 1935, 68, 82, 2023.

**Olivetoric Acid**

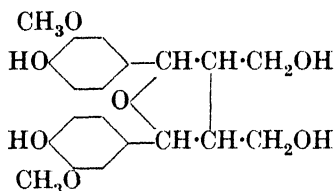
Occurs in lichens *Alectoria divergens* Nyl, *Parmelia olivetorum* Nyl, *Evernia olivetorina* Zopf. Cryst. from hot C<sub>6</sub>H<sub>6</sub>. M.p. 151°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

*6 : 3'-Di-Me ether* : *Me ester*,  $\text{C}_{29}\text{H}_{38}\text{O}_8$ . MW, 514. M.p. 123°.

*Me ester*:  $C_{27}H_{34}O_8$ . MW, 486. Needles. M.p.  $134^\circ$ .

Asahina, Fuzikawa, *Ber.*, 1935, **68**, 2026.  
Asahina, Asano, *Ber.*, 1932, **65**, 584.  
See also Hesse, *J. prakt. Chem.*, 1916, **94**, 227.

## Olivil



$C_{20}H_{24}O_7$  MW, 376

Occurs in olive-wood resin. Cryst.  $+ 1H_2O$  from  $H_2O$ . M.p.  $105^\circ$ , anhyd.  $142.5^\circ$ .  $[\alpha]_D^{25} - 127^\circ$ .

*Me ether*:  $C_{21}H_{26}O_7$ . MW, 390. M.p.  $238^\circ$ .

*Di-Me ether*:  $C_{22}H_{28}O_7$ . MW, 404. M.p.  $156^\circ$ .

*Et ether*:  $C_{22}H_{28}O_7$ . MW, 404. M.p.  $145^\circ$ .

*Di-Et ether*:  $C_{24}H_{32}O_7$ . MW, 432. M.p.  $182^\circ$ .

*Me-Et ether*:  $C_{23}H_{30}O_7$ . MW, 418. M.p.  $169^\circ$ .

*Dipropyl ether*:  $C_{26}H_{36}O_7$ . MW, 460. M.p.  $135.5^\circ$ .

Vanzetti, Dreyfuss, *Gazz. chim. ital.*, 1934, **64**, 381.

Dreyfuss, *Gazz. chim. ital.*, 1936, **66**, 96.

## Onocerin.

See Onocerol.

**Onocerol** (*Onocol, onocerin*)

$C_{26}H_{44}O_2$  MW, 288

A phytosterol occurring in *Ononis spinosa*, Linn. Prisms from EtOH. M.p.  $232^\circ$ . Sol. isoamyl alcohol. Spar. sol.  $Et_2O$ ,  $CHCl_3$ , AcOEt. Insol.  $H_2O$ . Sublimes.  $[\alpha]_D^{20} + 12.05^\circ$ .

*Diacetyl deriv.*: m.p.  $224^\circ$ .  $[\alpha]_D^{18} + 28.3^\circ$  in  $CHCl_3$ .

*Di-chloroacetyl deriv.*: m.p.  $238-44^\circ$ .  $[\alpha]_D^{19} + 115^\circ$  in  $CHCl_3$ .

*Dibenzoyl deriv.*: m.p.  $237-8^\circ$ .  $[\alpha]_D^{22} + 21.3^\circ$  in  $CHCl_3$ .

*Di-3:5-dinitrobenzoyl deriv.*: m.p.  $290-1^\circ$ .  $[\alpha]_D^{19} + 19.3^\circ$  in  $CHCl_3$ .

*Dianisoyl deriv.*: m.p.  $232-4^\circ$ .  $[\alpha]_D^{22} + 10^\circ$  in  $CHCl_3$ .

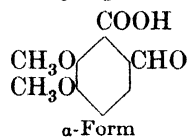
Schulze, *Z. physiol. Chem.*, 1936, **238**, 35.

Dieterle, Salomon, Gärtner, *Chem. Abstracts*, 1934, **28**, 4065.

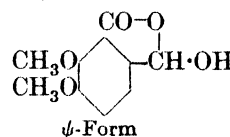
## Onocol.

See Onocerol.

**Opianic Acid** (5:6-Dimethoxy-o-aldehydobenzoic acid, 6-aldehydo-o-veratric acid, 5:6-dimethoxy-o-phthalaldehydic acid)



$\alpha$ -Form



$\psi$ -Form

$C_{10}H_{10}O_5$  MW, 210

Prisms from  $H_2O$ . M.p.  $150^\circ$  ( $146^\circ$ ). Sol. EtOH,  $Et_2O$ .  $k = 8.82 \times 10^{-4}$  at  $25^\circ$ . Ox.  $\rightarrow$  hemipinic acid. Red.  $\rightarrow$  meconin.

$\alpha$ -*Me ester*:  $C_{11}H_{12}O_5$ . MW, 224. Needles from EtOH. M.p.  $82-3^\circ$ . B.p.  $232-4^\circ/52$  mm.

*Diacetyl*: m.p.  $88-9^\circ$ . *Semicarbazone*: m.p.  $204^\circ$ .

$\psi$ -*Me ester*: m.p.  $105^\circ$ .

$\alpha$ -*Et ester*:  $C_{12}H_{14}O_5$ . MW, 238. M.p.  $64^\circ$ .

$\psi$ -*Et ester*: m.p.  $92-3^\circ$ .

$\psi$ -*Propyl ester*:  $C_{13}H_{16}O_5$ . MW, 252. M.p.  $103^\circ$ .

$\psi$ -*tert.-Amyl ester*:  $C_{15}H_{20}O_5$ . MW, 280. M.p.  $81^\circ$ .

$\alpha$ -*Benzyl ester*:  $C_{17}H_{16}O_5$ . MW, 300. M.p.  $82-3^\circ$ .

$\psi$ -*Benzyl ester*: m.p.  $94-5^\circ$ .

$\psi$ -*Chloride*:  $C_{10}H_9O_4Cl$ . MW, 228.5. Needles from  $C_6H_6$ . M.p.  $93-4^\circ$ .

*Anhydride*:  $C_{20}H_{18}O_9$ . MW, 402. M.p.  $234^\circ$ .

*Oxime*: needles. M.p.  $82^\circ$ .

*Semicarbazone*: m.p.  $187^\circ$ .

*Benzoylhydrazone*: m.p.  $227^\circ$  decomp.

Kanewskaja *et al.*, *Ber.*, 1936, **69**, 257.

Edwards, Perkin, Stoyale, *J. Chem. Soc.*, 1925, 197.

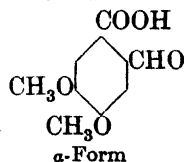
Kirpal, *Ber.*, 1927, **60**, 382.

Schorigin, Issagulan, Below, *Ber.*, 1931, **64**, 1931.

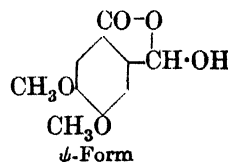
Wegscheider, Späth, *Monatsh.*, 1916, **37**, 277.

Rodionow, Fedorova, *Ber.*, 1926, **59**, 2949.

**m-Opianic Acid** (4:5-Dimethoxy-o-aldehydobenzoic acid, 6-aldehydo-veratric acid, 4:5-dimethoxy-o-phthalaldehydic acid)



$\alpha$ -Form



$\psi$ -Form

$C_{10}H_{10}O_5$

MW, 210

Needles from H<sub>2</sub>O. M.p. 185°. Sol. AcOH, hot Me<sub>2</sub>CO. Spar. sol. H<sub>2</sub>O, MeOH.

α-Me ester: C<sub>11</sub>H<sub>12</sub>O<sub>5</sub>. MW, 224. M.p. 93–5°. Anil: m.p. about 143°.

ψ-Me ester: m.p. 142–3°.

Anhydride: C<sub>20</sub>H<sub>18</sub>O<sub>9</sub>. MW, 402. M.p. about 230°.

Oxime: m.p. about 140°.

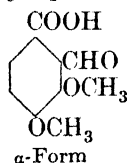
Semicarbazone: m.p. about 227°.

Phenylhydrazone: m.p. 228°.

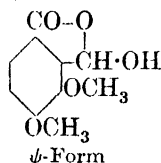
Vanzetti, Oliverio, Cavinato, *Gazz. chim. ital.*, 1931, **61**, 479.

Perkin, Stoye, *J. Chem. Soc.*, 1923, **123**, 3171.

ψ-Opianic Acid (3 : 4-Dimethoxy-o-aldehydobenzoic acid, 2-aldehydoveratric acid, 3 : 4-dimethoxy-o-phthalaldehydic acid)



α-Form



ψ-Form

C<sub>10</sub>H<sub>10</sub>O<sub>5</sub> MW, 210

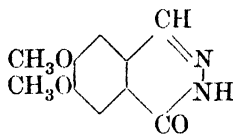
Needles from H<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>. M.p. 121–2°. Sol. hot H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether. Hot conc. KOH.Aq. → veratric acid.

Oxime: needles from H<sub>2</sub>O. M.p. 124° decomp.

Perkin, *J. Chem. Soc.*, 1890, **57**, 1064.

Chakravarti, Swaminathan, *Chem. Abstracts*, 1934, **28**, 6720.

Opiazone (6 : 7-Dimethoxyphthalazone)



C<sub>10</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub> MW, 206

Needles. M.p. anhyd. 166°. Sol. EtOH, AcOH, hot H<sub>2</sub>O.

N-Acetyl: m.p. 158–9°.

Liebermann, Bistrzycki, *Ber.*, 1893, **26**, 532.

Opsopyrrole.

See 4-Methyl-3-ethylpyrrole.

Optochine (*Dihydrocupreine ethyl ether*)

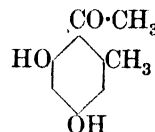
C<sub>21</sub>H<sub>28</sub>O<sub>2</sub>N<sub>2</sub> MW, 340

M.p. 80–4° (contains H<sub>2</sub>O and toluene). M.p. 123–8° (solvent free). Sol. ord. org. solvents.

Mod. sol. C<sub>6</sub>H<sub>6</sub>, toluene, ligroin. [α]<sub>D</sub><sup>20</sup> – 112.7° in EtOH ([α]<sub>D</sub><sup>20</sup> – 136.2° in EtOH).

Heidelberger, Jacobs, *J. Am. Chem. Soc.*, 1922, **44**, 1097.

Orcacetophenone (4 : 6-Dihydroxy-2-methylacetophenone, methyl 4 : 6-dihydroxy-o-tolyl ketone)



C<sub>9</sub>H<sub>10</sub>O<sub>3</sub> MW, 166

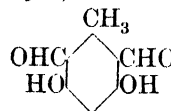
Needles from H<sub>2</sub>O. M.p. 159°. Sol. EtOH, Et<sub>2</sub>O, AcOEt, Me<sub>2</sub>CO. Mod. sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O, ligroin.

4-Me ether: C<sub>10</sub>H<sub>12</sub>O<sub>3</sub>. MW, 180. M.p. 79°.

6-Me ether: m.p. 150°.

Hoesch, *Ber.*, 1915, **48**, 1127.

α-Orcindialdehyde (4 : 6-Dihydroxy-2-methylisophthalaldehyde)

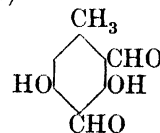


C<sub>9</sub>H<sub>8</sub>O<sub>4</sub> MW, 180

Needles from hot H<sub>2</sub>O. M.p. 117–19°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Mod. sol. hot H<sub>2</sub>O. Volatile in steam.

Tiemann, Helkenberg, *Ber.*, 1879, **12**, 1003.

β-Orcindialdehyde (2 : 6-Dihydroxy-4-methylisophthalaldehyde)

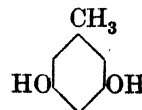


C<sub>9</sub>H<sub>8</sub>O<sub>4</sub> MW, 180

Cryst. from EtOH.Aq. M.p. 168°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, hot H<sub>2</sub>O.

Tiemann, Helkenberg, *Ber.*, 1879, **12**, 1004.

Orcinol (5-Methylresorcinol, 3 : 5-dihydroxy-toluene)



C<sub>7</sub>H<sub>8</sub>O<sub>2</sub> MW, 124

Cryst. + 1H<sub>2</sub>O from H<sub>2</sub>O, leaflets from CHCl<sub>3</sub>. (i) M.p. 106.5–108°. (ii) M.p. 107.5°. B.p.

287–90°. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. ligroin, pet. ether. Heat of comb.  $C_p$  824.72 Cal.

*Diacetyl*: m.p. 25°.

*Dibenzoyl*: m.p. 88°.

*Me ether*:  $C_8H_{10}O_2$ . MW, 138. M.p. 61–2°. B.p. 259°/755 mm., 130°/6.5 mm.  $D^{15}_D$  1.1106.  $n^{20}_D$  1.54734.

*Di-Me ether*:  $C_8H_{12}O_2$ . MW, 152. B.p. 244°, 102°/8 mm.  $D^{15}_D$  1.0478.  $n^{20}_D$  1.52342.

*Et ether*:  $C_9H_{12}O_2$ . MW, 152. B.p. 265–70°.

*Di-Et ether*:  $C_{11}H_{16}O_2$ . MW, 180. M.p. 16–16.5°. B.p. 251–2°/747.5 mm.

*Picrate*: m.p. 92°.

Schaum, *Ann.*, 1928, **462**, 207.

Missenden, *Chem. Age*, 1922, **7**, 709 (Review).

Walbaum, Rosenthal, *Ber.*, 1924, **57**, 771.

Vogt, Henninger, *Bull. soc. chim.*, 1874, **21**, 273.

### **$\beta$ -Orcinol.**

See 2 : 6-Dihydroxy-*p*-xylene.

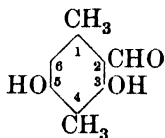
### **$\gamma$ -Orcinol.**

2 : 4-Dihydroxytoluene, *q.v.*

### **Orcinol-carboxylic Acid.**

See Orsellinic Acid and 3 : 5-Dihydroxy-*p*-toluic Acid.

**$\beta$ -Orcylaldehyde** ( *$\beta$ -Orcinaldehyde*, 3 : 5-dihydroxy-4-methyl-*o*-toluic aldehyde, 4 : 6-dihydroxy-2 : 5-dimethylbenzaldehyde)



$C_9H_{10}O_3$

MW, 166

Needles from EtOH. M.p. 166°.

*5-Me ether*: see Rhizonaldehyde.

Sonn, *Ber.*, 1931, **64**, 185.

Robertson, Stephenson, *J. Chem. Soc.*, 1930, 316.

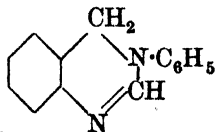
### **$\gamma$ -Orcylaldehyde.**

See Atranol, Addendum, Vol. I.

### **Orcylic Aldehyde.**

See 3 : 5-Dihydroxy-*o*-toluic Aldehyde.

**Orexine** (3-Phenyl-3 : 4-dihydroquinazoline)



$C_{14}H_{12}N_2$

MW, 208

Plates from  $Et_2O$ -ligroin. M.p. 94–6°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $H_2O$ .

*B, HCl, 2H\_2O*: m.p. 80°, anhyd. 221°.

*B, HCl, SnCl\_2*: m.p. 130–4°.

*B\_2, H\_2PtCl\_6*: m.p. 208°.

*B\_2, H\_2SO\_4, 2H\_2O*: m.p. 70°, anhyd. 140–3°.

*Methiodide*: m.p. 170°.

Paal, Busch, *Ber.*, 1889, **22**, 2686.

Kalle, D.R.P., 113,163, (*Chem. Zentr.*, 1900, II, 615).

### **Oripavine**

$C_{18}H_{21}O_3N$

MW, 299

Alkaloid from *Papaver orientalis*. Needles from EtOH. M.p. 200–1°. Sol.  $CHCl_3$ . Spar. sol. EtOH,  $Me_2CO$ . Insol.  $H_2O$ .

*B, HCl*: m.p. 244–5°.

*Methiodide*: m.p. 207–8°.

Konowalowa, Yunusoff, Orechhoff, *Ber.*, 1935, **68**, 2160.

### **Oroxidine**

$C_{15}H_{13}O_4N$

MW, 271

Needles from  $+ 1\frac{1}{2}H_2O$ , EtOH.Aq. M.p. 195°. Cryst. from ( $+ 1AcOH$  ?)  $AcOH$ . M.p. 142°.

Terasaka, *Chem. Abstracts*, 1932, **26**, 730.

### **Orixine**

$C_{18}H_{23}O_6N(C_{18}H_{21}O_6N)$

MW, 349 (347)

Alkaloid from root of *Orixu japonica*, Thunb. M.p. 152.5°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $AcOEt$ . Insol. pet. ether.  $[\alpha]^{25}_D + 83.29^\circ$  in  $CHCl_3$ .

*B, H, AuCl\_4*: decomp. at 155°.

See previous reference.

### **Orizabin.**

See Jalapin.

**Ornithine** (1 : 4-Diamino-*n*-valeric acid)

$H_2N \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot CH(NH_2) \cdot COOH$

$C_5H_{12}O_2N_2$

MW, 132

*dl.*

M.p. 140°. Sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ .  $[\alpha]^{25}_D + 11.5^\circ$ .

*B, 2HCl*:  $[\alpha]_D + 16.8^\circ$  in  $H_2O$ .

*B, H\_2PtCl\_6*: decomp. at 200–10°.

*Monosulphate*: decomp. at 234°.

*Acetate*: m.p. 161–2°.

1 : 4-*N*-Dibenzoyl: see Ornithuric Acid.

*Dipicrate*: decomp. at 208°.

*dl.*

*B, HCl*: m.p. 215°.

*B, HNO\_3*: m.p. 183°.

*B, H\_2SO\_4*: decomp. at 208°.

$B_2H_2SO_4$ : m.p. 213°.

Acetate: m.p. 163-4°.

Oxalate: m.p. 218°.

Picrolonate: m.p. 220-1° decomp.

Dipicrolonate: m.p. 235-6°.

4-N-Benzoyl: m.p. 285-8°.

Dipicrate: decomp. at 208° (m.p. 195°).

1-N-Me: 4-amino-1-methylamino-*n*-valeric acid.  $C_6H_{14}O_2N_2$ . MW, 146.  $B_2HAuCl_4$ : needles. M.p. 130-2°. Flavianate: m.p. 222-3°. 4-N-Benzoyl-1-N-p-toluenesulphonyl: needles from 70% EtOH. M.p. 185°. 1-N-p-Toluenesulphonyl: plates from  $H_2O$ . M.p. 214-19°. Prac. insol. EtOH,  $Et_2O$ , ligroin.

4-N-Me: 1-amino-4-methylamino-*n*-valeric acid.  $B_2HCl$ : m.p. about 215-25°.  $B_2HCl$ : m.p. 157°. Very sol.  $H_2O$ . 4-N-Benzoyl: m.p. 215°. Sol.  $H_2O$ . 1-N-p-toluenesulphonyl: decomp. at 245°. 4-N-Benzoyl-1-N-p-toluenesulphonyl: needles from AcOH. M.p. 188-9°. Picrate: decomp. at 205-6°.

Karrer, Escher, Widmer, *Helv. Chim. Acta*, 1926, **9**, 301.

Bergmann, Köster, *Z. physiol. Chem.*, 1926, **159**, 179.

Vickery, Cook, *J. Biol. Chem.*, 1931, **94**, 393.

Lutz, Jirgensons, *Ber.*, 1931, **64**, 1221.

Keimatsu, Sugawara, *Chem. Abstracts*, 1928, **22**, 1758.

Zimmermann, Canzanelli, *Z. physiol. Chem.*, 1933, **219**, 207.

Thomas, Kapfhammer, Flaschenträger, *Z. physiol. Chem.*, 1922, **124**, 75.

Boon, Robson, *Biochem. J.*, 1935, **29**, 2684.

### Ornithuric Acid (1 : 4-N-Dibenzoylornithine)

$C_6H_5 \cdot CO \cdot NH \cdot [CH_2]_3 \cdot CH(NH \cdot CO \cdot C_6H_5) \cdot COOH$

$C_{19}H_{20}O_4N_2$  MW, 340

*d*-.

Needles or plates from EtOH. M.p. 188-9°. Sol. AcOEt, hot EtOH. Spar. sol.  $H_2O$ . Insol.  $Et_2O$ .  $[\alpha]_D^{20} + 8.5^\circ$  in EtOH.Aq.

Me ester:  $C_{20}H_{22}O_4N_2$ . MW, 354. M.p. 145-6°.

Et ester:  $C_{21}H_{24}O_4N_2$ . MW, 368. M.p. 155°.

*l*-.

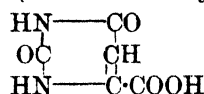
M.p. 189°.  $[\alpha]_D^{20} - 9.22^\circ$  in NaOH.Aq.

*dl*-.

Needles from EtOH. M.p. 187-8°.

See first and last references above.

### Orotic Acid (Uracil-4-carboxylic acid)



$C_5H_4O_4N_2$  MW, 156

Occurs in milk. Cryst. from  $H_2O$ . M.p. 345-6°.

Me ester:  $C_6H_6O_4N_2$ . MW, 170. M.p. 249°.

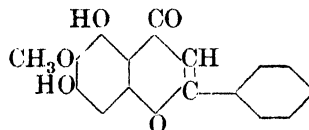
Et ester:  $C_7H_8O_4N_2$ . MW, 184. M.p. 188-9°.

Bachstetz, *Ber.*, 1931, **64**, 2683.

Hilbert, *J. Am. Chem. Soc.*, 1932, **54**, 2082.

Johnson, Schroeder, *J. Am. Chem. Soc.*, 1932, **54**, 2942.

### Oroxylin-A (5 : 7-Dihydroxy-6-methoxy-flarone)



$C_{16}H_{12}O_5$  MW, 284

Colouring matter of root bark of *Oroxylum indicum*, Vent. Yellow needles from EtOH. M.p. 231-2°.

Diacetyl: m.p. 131-2°.

7-Benzoyl: m.p. 210°.

7-Me ether:  $C_{17}H_{14}O_5$ . MW, 298. M.p. 155-6°. Platinichloride: m.p. 185-7° decomp.

5-Acetyl: m.p. 130-1°. 5-Benzoyl: m.p. 206-7°.

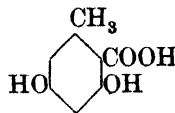
5 : 7-Di-Me ether:  $C_{18}H_{16}O_5$ . MW, 312. M.p. 165-6°.

Shah, Mehta, Wheeler, *J. Chem. Soc.*, 1936, 591.

### Orsellic Acid.

See Orsellinic Acid.

**Orsellinic Acid** (Orsellic acid, 3 : 5-dihydroxy-o-toluic acid, orcinol-2-carboxylic acid)



$C_8H_8O_4$  MW, 168

Needles +  $1H_2O$  from AcOH.Aq. M.p. 176° decomp. Sol. EtOH,  $Et_2O$ .  $k = 1.271 \times 10^{-4}$  at 25°.

Me ester:  $C_9H_{10}O_4$ . MW, 182. M.p. 140°.

Et ester:  $C_{10}H_{12}O_4$ . MW, 196. M.p. 132°.

Isoamyl ester:  $C_{13}H_{18}O_4$ . MW, 238. M.p. 76°.

Diacetyl: m.p. 142°. Chloride: m.p. 56-8°.

3-Me ether: see Isoevernic Acid.

5-Me ether : see Everninic Acid.

Di-Me ether : see under Everninic Acid.

5-Me-3-Et ether : see under Everninic Acid.

Hesse, *Ann.*, 1866, **139**, 35.

Hoesch, *Ber.*, 1913, **46**, 888.

Koller, *Monatsh.*, 1932, **61**, 147.

Sonn, *Ber.*, 1928, **61**, 926.

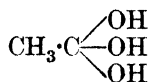
### p-Orsellinic Acid.

See 3 : 5-Dihydroxy-p-toluic Acid.

### Orthanilic Acid.

See Aniline-o-sulphonic Acid.

Orthoacetic Acid (1 : 1 : 1-Trihydroxyethane)



$\text{C}_2\text{H}_5\text{O}_3$

MW, 78

Tri-Me ester :  $\text{C}_5\text{H}_{12}\text{O}_3$ . MW, 120. B.p. 107–9°.  $D_4^{25}$  0.94375.  $n_D^{25}$  1.38585.

Di-Me : Et ester :  $\text{C}_6\text{H}_{14}\text{O}_3$ . MW, 134. B.p. 123–6°.  $D_4^{25}$  0.91915.  $n_D^{25}$  1.38885.

Me-di-Et ester :  $\text{C}_7\text{H}_{16}\text{O}_3$ . MW, 148. B.p. 135–6°.  $D_4^{25}$  0.90085.  $n_D^{25}$  1.39185.

Me-di-isobutyl ester :  $\text{C}_7\text{H}_{16}\text{O}_3$ . MW, 148. B.p. 205–6°.

Me-di-isoamyl ester :  $\text{C}_9\text{H}_{20}\text{O}_3$ . MW, 176. B.p. 219–23°.

Tri-Et ester :  $\text{C}_8\text{H}_{18}\text{O}_3$ . MW, 162. B.p. 144–6°.  $D_4^{25}$  0.8847.  $n_D^{25}$  1.39485.

Et-dipropyl ester :  $\text{C}_{10}\text{H}_{22}\text{O}_3$ . MW, 190. B.p. 190–4°.  $D_4^{25}$  0.87129.  $n_D^{25}$  1.40635.

Et-di-butyl ester :  $\text{C}_{12}\text{H}_{26}\text{O}_3$ . MW, 218. B.p. 220–5°.  $D_4^{25}$  0.86461.  $n_D^{25}$  1.41485.

Di-isobutyl ester :  $\text{C}_{10}\text{H}_{22}\text{O}_3$ . MW, 190. B.p. 207–8°.

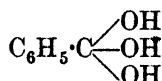
Tri-isobutyl ester :  $\text{C}_{14}\text{H}_{30}\text{O}_3$ . MW, 246. B.p. 217°.

Sah, *J. Am. Chem. Soc.*, 1928, **50**, 516;

*Chem. Abstracts*, 1933, **27**, 5729.

Sigmund, Herschdörfer, *Monatsh.*, 1931, **58**, 284.

### Orthobenzoic Acid ( $\omega$ -Trihydroxytoluene)



$\text{C}_7\text{H}_8\text{O}_3$

MW, 140

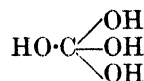
Tri-Et ester :  $\text{C}_{13}\text{H}_{20}\text{O}_3$ . MW, 224. B.p. 238–40°/747 mm.  $D_4^{20}$  0.9902.

Di-2-naphthyl ester anhydride : needles from  $\text{PhNO}_2$ . Does not melt below 350°.

Doebner, *Ann.*, 1890, **257**, 59.

Tschitschibabin, *Ber.*, 1905, **38**, 563.

### Orthocarbonic Acid (Tetrahydroxymethane)



$\text{CH}_4\text{O}_4$

MW, 80

Tetra-Me ester :  $\text{C}_5\text{H}_{12}\text{O}_4$ . MW, 136. M.p. –5.5°. B.p. 114°.  $D_{15}^{15.5}$  1.0232.  $n_D^{15}$  1.3864.

Tetra-Et ester :  $\text{C}_9\text{H}_{20}\text{O}_4$ . MW, 192. B.p. 158–9°, 62°/28 mm.  $D_4^{18.5}$  0.9197.  $n_D^{18.5}$  1.39354.

Tetrapropyl ester :  $\text{C}_{13}\text{H}_{28}\text{O}_4$ . MW, 248. B.p. 224.2°.

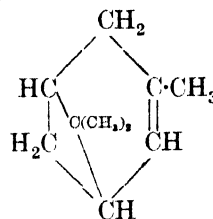
Tetra-isobutyl ester :  $\text{C}_{17}\text{H}_{36}\text{O}_4$ . MW, 304. B.p. 244.9°.

Hartel, *Ber.*, 1927, **60**, 1841.

Röse, *Ann.*, 1880, **205**, 250.

Wilke, *Z. anorg. allgem. Chem.*, 1921, **119**, 377.

### Orthodene



$\text{C}_{10}\text{H}_{16}$

MW, 136

Occurs in essential oil of *Orthodon lanceolatum*, Kudo. B.p. 168–70°/757 mm.  $D_4^{30}$  0.8430.  $n_D^{30}$  1.4670.  $[\alpha]_D^{25} + 32.6^\circ$ .

Fujita, *Chem. Abstracts*, 1934, **28**, 1470.

### Orthodonene

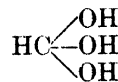
$\text{C}_{15}\text{H}_{24}$

MW, 204

Occurs in essential oil of *Orthodon lanceolatum*, Kudo. B.p. 254°/700 mm.  $D_4^{30}$  0.9017.  $n_D^{30}$  1.4947.  $[\alpha]_D^{14} - 13.28^\circ$ .

See previous reference.

### Orthoformic Acid (Trihydroxymethane)



$\text{CH}_4\text{O}_3$

MW, 64

Tri-Me ester :  $\text{C}_4\text{H}_{10}\text{O}_3$ . MW, 106. B.p. 103–5°.  $D_4^{20}$  0.9676.  $n_D^{20}$  1.3793.

Tri-Et ester :  $\text{C}_7\text{H}_{16}\text{O}_3$ . MW, 148. B.p. 145–7°, 60°/30 mm.  $D_4^{20}$  0.8909.  $n_D^{20}$  1.3922.

Di-Et : propyl ester :  $\text{C}_8\text{H}_{18}\text{O}_3$ . MW, 162. B.p. 165°/747 mm., 81°/30 mm.  $D_4^{20}$  0.8813.  $n_D^{20}$  1.3989.

Et-dipropyl ester :  $\text{C}_9\text{H}_{20}\text{O}_3$ . MW, 176. B.p. 184°/745 mm., 93°/30 mm.  $D_4^{22}$  0.8973.  $n_D^{20}$  1.4031.



*Tripropyl ester*:  $C_{10}H_{22}O_3$ . MW, 190. B.p.  $196-8^\circ$ ,  $190-1^\circ/745$  mm.,  $93^\circ/30$  mm.  $D_4^{20}$  0.8805.  $n_D^{20}$  1.4072.

*Dipropyl-isoamyl ester*:  $C_{12}H_{26}O_3$ . MW, 218. B.p.  $124-30^\circ/24$  mm.  $D_4^{23}$  0.8647.  $n_D^{20}$  1.415.

*Propyl-di-isoamyl ester*:  $C_{14}H_{30}O_3$ . MW, 246. B.p.  $140-7^\circ/30$  mm.  $D_4^{23}$  0.8626.  $n_D^{20}$  1.4194.

*Tri-isopropyl ester*: b.p.  $166-8^\circ$ .  $D_4^{20}$  0.8621.  $n_D^{20}$  1.4000.

*Tributyl ester*:  $C_{13}H_{28}O_3$ . MW, 232. B.p.  $245-7^\circ$ .  $D_4^{20}$  0.8693.  $n_D^{20}$  1.4180.

*Tri-isobutyl ester*: b.p.  $224-6^\circ$ .  $D_4^{20}$  0.8582.  $n_D^{20}$  1.4120.

*Tri-isoamyl ester*:  $C_{16}H_{34}O_3$ . MW, 274. B.p.  $267-9^\circ$ ,  $166^\circ/25$  mm.  $D_4^{20}$  0.8628.  $n_D^{20}$  1.4233.

*Triphenyl ester*:  $C_{19}H_{16}O_3$ . MW, 292. M.p.  $76-7^\circ$ . B.p.  $269-70^\circ/50-5$  mm. decomp. Sol.  $Et_2O$ ,  $CHCl_3$ , hot  $EtOH$ , hot  $C_6H_6$ .

*Tri-o-tolyl ester*:  $C_{22}H_{22}O_3$ . MW, 334. M.p.  $96^\circ$ .

*Tri-m-tolyl ester*: m.p.  $50^\circ$ .

*Tri-p-tolyl ester*: m.p.  $112^\circ$ .

*Trianilide*: trianilinomethane. M.p.  $138^\circ$ .  $B,3HCl$ : m.p.  $240^\circ$  decomp.

*Tri-o-toluidide*: m.p.  $150-1^\circ$ .  $B,3HCl$ : m.p.  $212-13^\circ$ .

*Tri-m-toluidide*: m.p.  $123^\circ$ .  $B,3HCl$ : m.p.  $221-2^\circ$ .

Giaccalone, *Gazz. chim. ital.*, 1932, **62**, 577.  
Post, Erickson, *J. Am. Chem. Soc.*, 1933, **55**, 3851 (*Bibl.*).

Driver, *J. Am. Chem. Soc.*, 1924, **46**, 2090.

Hunter, *J. Chem. Soc.*, 1924, **125**, 1392.

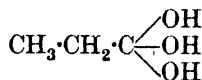
### Orthoform New.

See under 4-Hydroxy-*m*-aminobenzoic Acid.

### Orthoform Old.

See under 3-Hydroxy-*p*-aminobenzoic Acid.

**Orthopropionic Acid** (1 : 1 : 1-Trihydroxypropane)



$C_3H_6O_3$  MW, 92

*Tri-Et ester*:  $C_9H_{20}O_3$ . MW, 176. B.p.  $159-60^\circ$ .

Kodak-Pathé, F.P., 712,995, (*Chem. Abstracts*, 1932, **26**, 1531).

Sigmund, Herschdörfer, *Monatsh.*, 1931, **58**, 282.

### Oryzanin.

See Vitamin  $B_1$ .

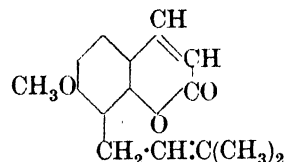
### Oscine.

See Scopoline.

### Osotriazole.

See 1 : 2 : 3-Triazole.

### Osthol



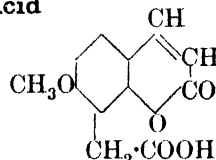
$C_{15}H_{16}O_3$  MW, 244

Occurs in root of *Imperatoria ostruthium*, Linn. Needles from  $EtOH$ . Aq. M.p.  $83-4^\circ$  ( $85^\circ$ ). B.p.  $145-50^\circ$ . Sol.  $EtOH$ ,  $MeOH$ ,  $CHCl_3$ ,  $Me_2CO$ ,  $AcOEt$ . Insol.  $H_2O$ , pet. ether.

Späth, Holzen, *Ber.*, 1934, **67**, 264.

Yamashita, *Bull. Chem. Soc. Japan*, 1933, **8**, 276.

### Ostholic Acid



$C_{12}H_{10}O_5$  MW, 234

Needles from  $AcOEt$ . M.p.  $254-5^\circ$ .

*Me ester*:  $C_{13}H_{12}O_5$ . MW, 248. M.p.  $155^\circ$ .

Späth, Pesta, *Ber.*, 1933, **66**, 759.

### Ostreastanol.

See Sitostanol.

### Ostreasterol-1

$C_{29}H_{48}O$  MW, 412

Occurs in oysters. M.p.  $142-3^\circ$ .  $[\alpha]_D^{20}$   $-43.57^\circ$  in  $CHCl_3$ .

*Acetyl deriv.*: m.p.  $134.5^\circ$ .  $[\alpha]_D^{20}$   $-45.95^\circ$  in  $CHCl_3$ .

*Propionyl deriv.*: m.p.  $113-14^\circ$ .

*Benzoyl deriv.*: m.p.  $145-7^\circ$ .

Bergmann, *J. Biol. Chem.*, 1934, **104**, 317, 553.

### Ostreasterol-2

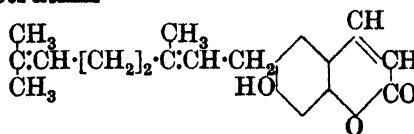
$C_{28}H_{46}O_2$  MW, 416

Plates from  $EtOH$ . M.p.  $122^\circ$ .

*Acetyl deriv.*: prisms. M.p.  $104^\circ$ .  $[\alpha]_D^{21}$   $-15.9^\circ$ .

See previous reference.

### Ostruthin



$C_{19}H_{22}O_3$  MW, 298

Occurs in root of *Imperatoria ostruthium*, Linn. Cryst. from EtOH.Aq. M.p. 119° (117°). Sol. CHCl<sub>3</sub>, AcOEt, hot EtOH. Insol. H<sub>2</sub>O, pet. ether.

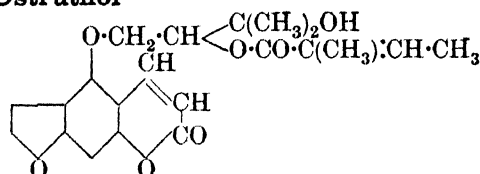
Acetyl deriv.: m.p. 80°.

Me ether: C<sub>20</sub>H<sub>24</sub>O<sub>3</sub>. MW, 312. M.p. 55-55.5°.

Späth, Klager, *Ber.*, 1934, **67**, 859.

Butenandt, Marten, *Ann.*, 1932, **495**, 197.

### Ostruthol



Probable structure

C<sub>21</sub>H<sub>22</sub>O<sub>7</sub>

MW, 386

Occurs in *Imperatoria ostruthium*, Linn. Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 136-7°. B.p. 225°/0.015 mm. [α]<sub>D</sub><sup>15</sup> - 18.3° in Py.

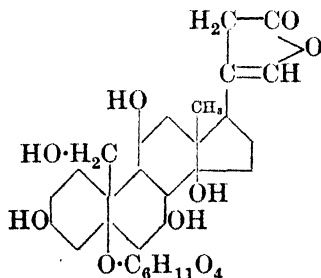
Acetyl deriv.: m.p. 125°.

Späth, Christiani, *Ber.*, 1933, **66**, 1150.

### Osyrtrin.

See Rutin.

### Ouabain (Acocantherin, g-strophanthin)



Suggested structure

C<sub>29</sub>H<sub>44</sub>O<sub>12</sub>

MW, 584

Glucoside occurring in *Acocanthera oabaio*. M.p. 180° (indefinite). Hyd. → rhamnose + acocanthic acid lactone.

Jacobs, Bigelow, *J. Biol. Chem.*, 1933, **101**, 15.

Schwurtze, Hann, Keenan, *Chem. Abstracts*, 1930, **24**, 917.

Moir, *Chem. Abstracts*, 1924, **18**, 1825.

Richaud, *Chem. Abstracts*, 1922, **16**, 314.

Catilloni, *Chem. Abstracts*, 1918, **12**, 741.

Klein, *Chem. Abstracts*, 1914, **8**, 988.

Arnaud, *Compt. rend.*, 1888, **107**, 1162.

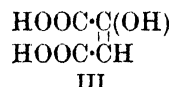
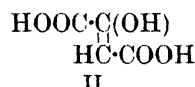
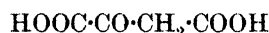
Fieser, Newman, *J. Biol. Chem.*, 1936, **114**, 705.

Dict. of Org. Comp.—III.

### Ovoflavine.

See Lactoflavine.

**Oxalacetic Acid** (*Ketosuccinic acid, hydroxy-fumaric acid, hydroxymaleic acid*)



C<sub>4</sub>H<sub>4</sub>O<sub>5</sub>

MW, 132

#### II.

Cryst. from Me<sub>2</sub>CO-C<sub>6</sub>H<sub>6</sub>. M.p. 184°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Heat of comb. C<sub>p</sub> 274.10 Cal., C<sub>v</sub> 275.78 Cal. *k* = 2.76 × 10<sup>-3</sup> at 17°. Py at 50° → III.

#### III.

Cryst. from Me<sub>2</sub>CO-C<sub>6</sub>H<sub>6</sub>. M.p. 152°. Sol. EtOH, Me<sub>2</sub>CO, AcOEt. Spar. sol. Et<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, ligroin. Heat of comb. C<sub>p</sub> 284.9 Cal., C<sub>v</sub> 286.58 Cal. *k* = 2.505 × 10<sup>-3</sup> at 17°.

#### I.

A, 2NH<sub>3</sub>: m.p. 75-7° decomp.

A, (CO(NH<sub>2</sub>)<sub>2</sub>)<sub>2</sub>: m.p. 124° decomp.

Di-brucine salt: m.p. 163-6°. Esters and salts are probably of I.

Di-Me ester: C<sub>6</sub>H<sub>8</sub>O<sub>5</sub>. MW, 160. M.p. 74° (77°). Me ether: C<sub>7</sub>H<sub>10</sub>O<sub>5</sub>. MW, 174. B.p. 228-30°.

1-Et ester: C<sub>6</sub>H<sub>8</sub>O<sub>5</sub>. MW, 160. M.p. 102-3°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>.

Di-Et ester: C<sub>8</sub>H<sub>12</sub>O<sub>5</sub>. MW, 188. B.p. 131-2°/24 mm. D<sub>4</sub><sup>20</sup> 1.130-1.132. n<sub>D</sub><sup>15.6</sup> 1.45614.

Semicarbazone: m.p. 162°. p-Chlorophenylhydrazine: m.p. 119-20°. p-Tolylhydrazine: m.p. 105-6°.

1-Me: 2-Et ester: C<sub>7</sub>H<sub>10</sub>O<sub>5</sub>. MW, 174. B.p. 130°/22 mm., 124°/16 mm. Cu Salt: green prisms from EtOH. M.p. 134-5°.

2-Me: 1-Et ester: b.p. 110°/13 mm. Cu salt: green needles. M.p. 165-6° (173-4° anhyd.).

2-Et ester: 1-nitrile: oxime: isonitrososuccinic acid Et ester nitrile. M.p. 104°. Acetyl deriv.: m.p. 146°.

Di-isoamyl ester: C<sub>14</sub>H<sub>24</sub>O<sub>5</sub>. MW, 272. B.p. 167°/23 mm. Cu salt: green needles from EtOH. M.p. 83-5°.

Diamide: C<sub>4</sub>H<sub>6</sub>O<sub>3</sub>N<sub>2</sub>. MW, 130. M.p. 180° decomp.

*Anhydride*: *acetyl deriv.*: acetoxymaleic anhydride. M.p. 89–91°.

*Oxime*: isonitrososuccinic acid. (i) M.p. 126° decomp. (ii) M.p. 88° decomp.

2:4-Dinitrophenylhydrazone: m.p. 211° decomp.

Diels, Meyer, *Ann.*, 1934, 513, 139.

U.S. Industrial Alcohol Co., U.S.P., 1,948,201, (*Chem. Abstracts*, 1934, 28, 2730).

Blanchetière, *Compt. rend.*, 1916, 163, 206.

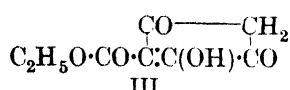
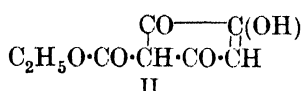
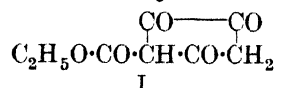
Hantzsch, *Ber.*, 1915, 48, 1407.

Gault, *Compt. rend.*, 1914, 158, 711.

Fenton, Wilks, *J. Chem. Soc.*, 1912, 101, 1570.

Meyer, *Ber.*, 1912, 45, 2860.

### Oxalacetoacetic Ethyl Ester



$\text{C}_8\text{H}_8\text{O}_5$  MW, 184

(a) Yellow needles from  $\text{Me}_2\text{CO}$ . M.p. 145–50° decomp. Sol.  $\text{H}_2\text{O}$ , hot  $\text{EtOH}$ , hot  $\text{AcOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ , ligroin.

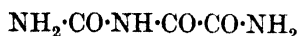
*Phenylhydrazone*: m.p. 184–6° decomp.

*Di-phenylhydrazone*: m.p. 198–202°.

(b) Yellow leaflets from  $\text{AcOEt}$ . M.p. 105–10°. Sol.  $\text{H}_2\text{O}$ . Mod. sol. ord. org. solvents.

Wislicenus, Schollkopf, *J. prakt. Chem.*, 1917, 95, 269.

### Oxalan (Oxaluramide)



$\text{C}_3\text{H}_5\text{O}_3\text{N}_3$  MW, 131

Cryst. Does not melt below 310°. Insol.  $\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{SO}_4$ , reprecipitated by  $\text{H}_2\text{O}$ .

Schenck, *Ber.*, 1905, 38, 459.

Seemann, *Z. physik. Chem.*, 1905, 44, 244.

### Oxalic Acid



$\text{C}_2\text{H}_2\text{O}_4$

MW, 90

Occurs in many plants. Cryst. +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. 101.5°, anhyd. 189.5°. Dehydrates on heating and sublimes at 150–60°. Sol. 10.5 parts  $\text{H}_2\text{O}$  at 15°. Mod. sol.  $\text{EtOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ .  $\text{Ca}^{++}$  and  $\text{Hg}^+$  salts very spar. sol.  $\text{H}_2\text{O}$ .  $k$  (first) =  $3.4\text{--}3.6 \times 10^{-2}$  at 25°.  $D_4^{18.5}$  1.653. Heat of comb.  $C_p$  60.2 Cal.,  $C_v$  61.1 Cal.

*Me ester*:  $\text{C}_3\text{H}_4\text{O}_4$ . MW, 104. B.p. 108–9°.

*Di-Me ester*: see Dimethyl oxalate.

*Me-Et ester*:  $\text{C}_5\text{H}_8\text{O}_4$ . MW, 132. B.p. 173.7°.  $D_0^{20}$  1.5505.

*Et ester*:  $\text{C}_4\text{H}_6\text{O}_4$ . MW, 118. B.p. 117°/15 mm.  $D_4^{20}$  1.2175. *Hydrazide*: m.p. 52–3° decomp.

*Di-Et ester*: see Diethyl oxalate.

*Ethylene ester*:  $\text{C}_4\text{H}_4\text{O}_4$ . MW, 116. M.p. 144°. Readily polymerises.

*Propyl ester*:  $\text{C}_5\text{H}_8\text{O}_4$ . MW, 132. B.p. 118–19°/13 mm.

*Dipropyl ester*:  $\text{C}_8\text{H}_{14}\text{O}_4$ . MW, 174. M.p. –46.3°. B.p. 214–15° (211–12°).  $D_4^{20}$  1.01693.  $n_D^{20}$  1.4168.

*Isopropyl ester*:  $\text{C}_5\text{H}_8\text{O}_4$ . MW, 132. B.p. 111°/13 mm.  $D_4^{20}$  1.1657.

*Di-isopropyl ester*:  $\text{C}_8\text{H}_{14}\text{O}_4$ . MW, 174. B.p. 189° (193–4°).  $D_4^{20}$  1.00097.  $n_D^{20}$  1.4100.

*Dibutyl ester*:  $\text{C}_{10}\text{H}_{18}\text{O}_4$ . MW, 202. F.p. –29.6°. B.p. 247–9°.  $D_4^{20}$  0.98732.  $n_D^{20}$  1.4234.

*Di-isobutyl ester*: b.p. 229–31°.  $D_4^{20}$  0.97373.  $n_D^{20}$  1.4180.

*Di-n-amyl ester*:  $\text{C}_{12}\text{H}_{22}\text{O}_4$ . MW, 230. M.p. –12.8°. B.p. 154.1°/14.8 mm.  $D^{20}$  0.9722.  $n_D^{20}$  1.4302.

*Di-isoamyl ester*: b.p. 267–8°.

*Di-n-hexyl ester*:  $\text{C}_{14}\text{H}_{26}\text{O}_4$ . MW, 258. M.p. –9.0°. B.p. 135.6°/2.5 mm.  $D^{20}$  0.9523.  $n_D^{20}$  1.4331.

*Di-n-heptyl ester*:  $\text{C}_{16}\text{H}_{30}\text{O}_4$ . MW, 286. M.p. 12.5°. B.p. 142–4°/2 mm.  $D^{20}$  0.9393.  $n_D^{20}$  1.4372.

*Di-n-nonyl ester*:  $\text{C}_{18}\text{H}_{34}\text{O}_4$ . MW, 314. M.p. 11.2°. B.p. 167–9°/3 mm.  $D^{20}$  0.9293.  $n_D^{20}$  1.4404.

*Dimyricyl ester*:  $\text{C}_{62}\text{H}_{122}\text{O}_4$ . MW, 930. M.p. 91°.

*Diallyl ester*:  $\text{C}_8\text{H}_{10}\text{O}_4$ . MW, 170. B.p. 206–7°/754 mm.

*Dicyclohexyl ester*:  $\text{C}_{14}\text{H}_{22}\text{O}_4$ . MW, 254. M.p. 42°.

*Diphenyl ester*:  $\text{C}_{14}\text{H}_{10}\text{O}_4$ . MW, 242. M.p. 134°.

*Di-o-nitrophenyl ester*:  $\text{C}_{14}\text{H}_8\text{O}_8\text{N}_2$ . MW, 332. M.p. 185°.

*Di-o-tolyl ester*:  $C_{16}H_{14}O_4$ . MW, 270. M.p. 90–1°.

*Di-m-tolyl ester*: m.p. 105°.

*Di-p-tolyl ester*: m.p. 148°.

*Di-p-nitrobenzyl ester*:  $C_{16}H_{12}O_8N_2$ . MW, 360. M.p. 204°.

*Me ester chloride*:  $C_3H_3O_3Cl$ . MW, 122.5. B.p. 125° (118–20°).  $D_4^{20}$  1.33163.

*Et ester chloride*:  $C_4H_5O_3Cl$ . MW, 136.5. B.p. 135°, 30°/10 mm.  $D_4^{20}$  1.2226.

*Propyl ester chloride*:  $C_5H_7O_3Cl$ . MW, 150.5. B.p. 156–8°, 50°/12 mm.  $D_4^{20}$  1.16697.

*Isobutyl ester chloride*:  $C_6H_9O_3Cl$ . MW, 164.5. B.p. 163–5°, 52°/10 mm.  $D_4^{20}$  1.11532.

*Isoamyl ester chloride*:  $C_7H_{11}O_3Cl$ . MW, 178.5. B.p. 183–5°, 68°/10 mm.  $D_4^{20}$  1.09312.

*Dichloride*: see Oxalyl chloride.

*Monoamide*: see Oxamic Acid.

*Diamide*: see Oxamide.

*Amide-nitrile*: see under Cyanoformic Acid.

*Mononitrile*: see Cyanoformic Acid.

*sym.-Dimethylamide*: see *sym.-Dimethyl-oxamide*.

*unsym.-Dimethylamide*: see Dimethyloxamic Acid.

*Amide-nitrile*: see under Cyanoformic Acid.

*Dinitrile*: see Cyanogen.

*Methylamide*: see Methyloxamic Acid.

*Monoanilide*: see Oxanilic Acid.

*Nitroanilide*: see Nitro-oxanilic Acid.

*Dianilide*: see Oxanilide.

*Di-o-nitroanilide*: yellow plates from aniline. M.p. 331° part. decomp.

*Di-m-nitroanilide*: needles from aniline. M.p. 309–10°.

*Di-p-nitroanilide*: yellowish needles from aniline. M.p. 358–9° (260°).

*Hydrazide*: does not melt below 300°.

*Dihydrazide*: m.p. 241° decomp. *N:N'-Di-acetyl*: m.p. 276°. *Hexa-acetyl*: m.p. 156–8°.

Johnson, Partington, *J. Chem. Soc.*, 1930, 1510.

Semenov, Shagalov, Astrakhantzev, *Chem. Abstracts*, 1935, 29, 6882.

Chrzaszcz, Zakomorzy, *Biochem. Z.*, 1935, 279, 64.

Skinner, *J. Am. Chem. Soc.*, 1933, 55, 2036.

Rakusin, *Chem.-Ztg.*, 1931, 55, 128.

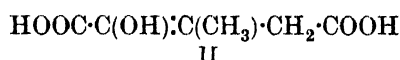
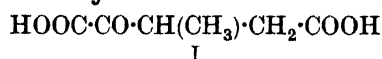
Jewel, Butts, *J. Am. Chem. Soc.*, 1931, 53, 3560.

Sah, Chen, *ibid.*, 3901.

Mugdan, Sint, D.R.P., 606,774, (*Chem. Abstracts*, 1935, 29, 3691).

du Pont, U.S.P., 1,948,441, (*Chem. Abstracts*, 1934, 28, 2728).

## 2-Oxalobutyric Acid

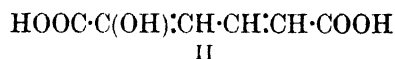
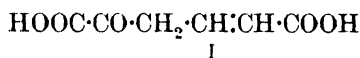


$C_6H_8O_5$  MW, 160

*Di-Et ester*:  $C_{10}H_{16}O_5$ . MW, 216. B.p. 163°/5 mm.

Feist, Brewer, *Ann.*, 1922, 428, 68.

**Oxalocrotonic Acid** (1-Hydroxymuconic acid)



$C_6H_6O_5$  MW, 158

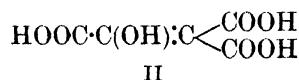
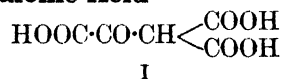
Yellow cryst. M.p. about 190° decomp. Sol. EtOH, AcOH, H·COOH. Spar. sol.  $H_2O$ . Insol.  $C_6H_6$ ,  $CHCl_3$ .

Lapworth, *J. Chem. Soc.*, 1901, 79, 1279.

## Oxalodiacetic Acid.

See Ketipic Acid.

## Oxalomalonic Acid



$C_5H_4O_7$  MW, 176

*Tri-Me ester*:  $C_8H_{10}O_7$ . MW, 218. Needles from  $Et_2O$ . M.p. 49–50°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. EtOH.

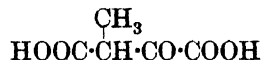
*Tri-Et ester*:  $C_{11}H_{16}O_7$ . MW, 260. B.p. 220°/10 mm.  $D_4^{20}$  1.153.  $n_D^{20}$  1.4468.

Auwers, Auffenberg, *Ber.*, 1918, 51, 1103.

## Oxalomethylaniline.

See N-Methyloxanilic Acid.

**1-Oxalopropionic Acid** (*Methyloxalacetic acid*)



$C_5H_6O_5$  MW, 146

*Di-Et ester*:  $C_9H_{14}O_5$ . MW, 202. Liq. B.p. 137–8°/23 mm., 114–16°/10 mm. Misc. with EtOH,  $Et_2O$ . Insol.  $H_2O$ . Decomp. on standing. Alc.  $FeCl_3 \rightarrow$  intense red col.

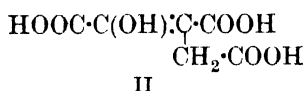
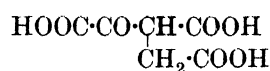
*Monoamide*:  $C_5H_7O_4N$ . MW, 145. Not isolated. *Phenyldiazone*: m.p. 99–100°.

*Mononitrile*:  $C_5H_5O_3N$ . MW, 127. Yellow cryst. from  $Et_2O$ -pet. ether. M.p.  $207-8^\circ$ . Sol.  $H_2O$ . Insol.  $C_6H_6$ . Reacts acid. Alc.  $FeCl_3 \rightarrow$  red col.

Wislicenus, Arnold, *Ann.*, 1888, **246**, 329.

Wislicenus, Silberstein, *Ber.*, 1910, **43**, 1829.

### Oxalosuccinic Acid



$C_6H_6O_7$  MW, 190

*Tri-Et ester*:  $C_{12}H_{18}O_7$ . MW, 274. B.p.  $170-5^\circ/12-13$  mm. *Phenylhydrazone*: decomp. at  $85^\circ$ .

Wislicenus, Waldmüller, *Ber.*, 1911, **44**, 1564.

### Oxaluramide.

See Oxalan.

### Oxaluric Acid (*Mono-oxalylurea*)



$C_3H_4O_4N_2$  MW, 132

Cryst. Decomp. at  $208-10^\circ$ . Sol.  $H_2O$ . Spar. sol. ord. org. solvents. Heat of comb. 207.7 Cal. Hot  $H_2O \rightarrow$  oxalic acid + urea.

*Me ester*:  $C_4H_6O_4N_2$ . MW, 146. M.p.  $192^\circ$  decomp.

*Et ester*:  $C_5H_8O_4N_2$ . MW, 160. M.p.  $177-8^\circ$  decomp.

$\omega$ -N-Formyl: m.p.  $175^\circ$  decomp.

Amide: see Oxalan.

Hydrazide: m.p.  $198^\circ$  decomp.

Fosse, Thomas, Graeve, *Compt. rend.*, 1935, **200**, 1260.

Biltz, Schauder, *J. prakt. Chem.*, 1923, **106**, 147.

### Oxalyl bromide



$C_2O_2Br_2$  MW, 216

Green liq. M.p.  $-19.5^\circ$ . B.p.  $102-3^\circ/720$  mm.,  $16-17^\circ/10$  mm.

Staudinger, Anthes, *Ber.*, 1913, **46**, 1431.

### Oxalylcarbanilide.

See Diphenylparabanic Acid.

### Oxalyl chloride



$C_2O_2Cl_2$  MW, 127

Needles from  $Et_2O$  or pet. ether at  $-80^\circ$ . M.p.  $-12^\circ$ . B.p.  $63.5-64^\circ/763$  mm.  $D_4^{25}$  1.4884.  $n_D^{25}$  1.434.

Staudinger, *Ber.*, 1908, **41**, 3563; D.R.Ps., 216,918-19, (*Chem. Abstracts*, 1910, **4**, 1087).

Jones, Tasker, *Proc. Chem. Soc.*, 1908, **24**, 271.

Giua, *Chem. Abstracts*, 1925, **19**, 1245.

### Oxalyl diacetic Acid.

See Ketipic Acid.

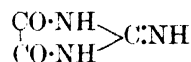
### Oxalyl-diacetophenone.

See Diphenacyl Diketone.

### Oxalyl dimethylurea.

See Dimethylparabanic Acid.

### Oxalylguanidine



$C_3H_3O_2N_3$  MW, 113

Prisms from  $H_2O$ . M.p.  $266-8^\circ$  decomp. (sealed tube). Sol.  $H_2O \rightarrow$  oxalic acid + guanidine slowly. Insol. EtOH.

Traube, *Ber.*, 1893, **26**, 2552.

### Oxalylmethylurea.

See Methylparabanic Acid.

### Oxalylurea.

See Oxaluric Acid and Parabanic Acid.

### Oxamethane.

See under Oxamic Acid.

**Oxamic Acid** (*Oxaminic acid*, *oxalic acid monoamide*)



$C_2H_3O_3N$  MW, 89

Cryst. from  $H_2O$ . M.p.  $210^\circ$  decomp. Spar. sol.  $H_2O$ . Insol. EtOH,  $Et_2O$ . Heat of comb.  $C_p$  132.0 Cal.,  $C_v$  129.5 Cal.

*Me ester*:  $C_3H_5O_3N$ . MW, 103. M.p.  $122-3^\circ$ .

*Et ester*: oxamethane.  $C_4H_7O_3N$ . MW, 117. M.p.  $114-15^\circ$ . N-Acetyl: m.p.  $53.5-54.5^\circ$ .

*Pentachloroethyl ester*:  $C_4H_2O_3NCl_5$ . MW, 289.5. M.p.  $134^\circ$ .

*Propyl ester*:  $C_5H_9O_3N$ . MW, 131. M.p.  $90-2^\circ$ .

*Isopropyl ester*: m.p.  $86-7^\circ$ .

*Butyl ester*:  $C_6H_{11}O_3N$ . MW, 145. M.p.  $82-4^\circ$ .

*Isobutyl ester*: m.p.  $89-90^\circ$ .

*Isoamyl ester*:  $C_7H_{13}O_3N$ . MW, 159. M.p. 92–3°.

*Amide*: see Oxamide.

*Hydrazide*: semioxamazide. M.p. 220–1° decomp. (223–4°).

*Diacetyl*:  $H_2N \cdot CO \cdot CO \cdot NH \cdot N(CO \cdot CH_3)_2$ . M.p. 184–5°.

*N-Me*: see Methyloxamic Acid.

*N-Di-Me*: see Dimethyloxamic Acid.

*Nitroanilide*: see under Nitro-oxanilic Acid.

Sah, Chen, *J. Am. Chem. Soc.*, 1931, **53**, 3901.

Oelkers, *Ber.*, 1889, **22**, 1569.

Weddige, *J. prakt. Chem.*, 1874, **10**, 196.

**Oxamide** (*Oxalic acid diamide*)



$C_2H_4O_2N_2$  MW, 88

Needles. Spar. sol. EtOH, hot  $H_2O$ . Decomp. above 320°. Heat of comb.  $C_p$  203.3 Cal.

N: *N'*-Dipropionyl: m.p. 216° decomp.

N: *N'*-Dibutyryl: m.p. 197°.

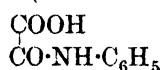
N: *N'*-Di-isobutyryl: m.p. 160°.

Kasiwagi, *Bull. Chem. Soc. Japan*, 1926, **1**, 67.

Langebeck, *Ann.*, 1929, **469**, 16.

Bucher, Canadian P., 173,369, (*Chem. Abstracts*, 1918, **12**, 156); U.S.P., 1,194,354, (*Chem. Abstracts*, 1916, **10**, 2500).

**Oxanilic Acid** (*Oxalic acid monoanilide*)



$C_8H_7O_3N$ . MW, 165

Needles from  $C_6H_6$ . M.p. 148–9° (149–50°). Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Mod. sol. hot  $H_2O$ . Spar. sol.  $C_6H_6$ , ligroin.  $k = 1.21 \times 10^{-2}$  at 25°. Heat of comb.  $C_p$  863.1 Cal.

*Me ester*:  $C_9H_9O_3N$ . MW, 179. M.p. 114° (111–12°).

*Et ester*:  $C_{10}H_{11}O_3N$ . MW, 193. M.p. 66–7°. Heat of comb.  $C_p$  1191.2 Cal. *N-Acetyl*: m.p. 64–5°.

*Propyl ester*:  $C_{11}H_{13}O_3N$ . MW, 207. M.p. 92°.

*Isopropyl ester*: m.p. 52°.

*Isobutyl ester*:  $C_{12}H_{15}O_3N$ . MW, 221. M.p. 85°.

*Isoamyl ester*:  $C_{13}H_{17}O_3N$ . MW, 235. M.p. 50°.

*m-Tolyl ester*:  $C_{15}H_{13}O_3N$ . MW, 255. M.p. 94°.

*p-Tolyl ester*: m.p. 132°.

*Chloride*:  $C_8H_6O_2NCl$ . MW, 183.5. M.p. 82.5°.

*Amide*:  $C_8H_8O_2N_2$ . MW, 164. M.p. 228° (224–5°).

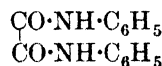
*Nitrile*:  $C_8H_6ON_2$ . MW, 146. M.p. 128° decomp.

*Anilide*: see Oxanilide.

Aschan, *Ber.*, 1890, **23**, 1820.

Stobbe, Knebel, *Ber.*, 1921, **54**, 1216.

**Oxanilide** (sym.-Diphenyloxamide, oxalic dianilide)



$C_{14}H_{12}O_2N_2$  MW, 240

Leaflets from  $C_6H_6$  or  $PhNO_2$ . M.p. 254° (252–3°). B.p. above 360°. Sol.  $C_6H_6$ . Spar. sol. hot EtOH. Insol.  $H_2O$ ,  $Et_2O$ . Heat of comb.  $C_p$  1665.4 Cal. Forms a *N*-Na deriv.

*N-Acetyl*: m.p. 197–8°.

N: *N'*-Diacetyl: m.p. 208–9° decomp.

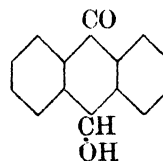
N: *N'*-Dibutyryl: m.p. 156°.

N: *N'*-Dibenzoyl: m.p. 212–13° decomp. (210°).

Bornwater, *Rec. trav. chim.*, 1912, **31**, 108.

Macullum, *J. Soc. Chem. Ind.*, 1923, **42**, 468r.

**Oxanthranol** (10-Hydroxyanthrone, oxanthrone)



$C_{14}H_{10}O_2$  MW, 210

Keto form of anthrahydroquinone. Colourless cryst. M.p. 167° decomp. Sols. do not fluoresce. Stable in air. Insol. cold alkalis. Sol. hot alc. alkalis, isomerising to anthrahydroquinone.  $Zn + AcOH \rightarrow$  anthranol.

*Acetyl*: m.p. 108–9°.

*Me ether*: 10-methoxyanthrone.  $C_{15}H_{12}O_2$ . MW, 224. M.p. 102.5° (98°).

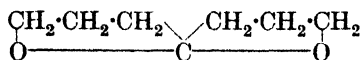
Meyer, *Ann.*, 1911, **379**, 60, 77.

**Oxanthrone.**

See Oxanthranol.

**Oxeserolene.**

See Hydroxyxeserolene.

**Oxetone** $\text{C}_7\text{H}_{12}\text{O}_2$ 

MW, 128

F.p. below  $-17^\circ$ . B.p.  $159.4^\circ$ . Mod. sol. cold  $\text{H}_2\text{O}$ . Spar. sol. hot  $\text{H}_2\text{O}$ . Spar. sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Reduces Tollen's reagent.

Fittig, Ström, *Ann.*, 1892, **267**, 197.

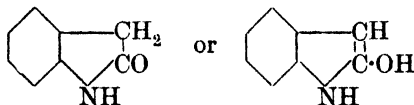
Granichstädten, Werner, *Monatsh.*, 1901, **22**, 333.

**Oximino-**

See also Isonitroso-

**Oximinoadipic Acid.**

See under 1-Ketoadipic Acid.

**Oxindole (2-Hydroxyindole)** $\text{C}_8\text{H}_7\text{ON}$ 

MW, 133

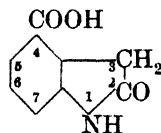
Needles from  $\text{H}_2\text{O}$ . M.p.  $127^\circ$ . B.p.  $227^\circ/23$  mm. Sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Reduces Tollen's reagent.

N-Acetyl: m.p.  $126^\circ$ .

Stollé, *J. prakt. Chem.*, 1930, **128**, 1.

Heller, *Ber.*, 1916, **49**, 2775.

Marschalk, *J. prakt. Chem.*, 1914, **88**, 234.

**Oxindole-4-carboxylic Acid** $\text{C}_9\text{H}_7\text{O}_3\text{N}$ 

MW, 177

Yellow cryst. Decomp. above  $280^\circ$ .

Braun, Hahn, *Ber.*, 1923, **56**, 2345.

**Oxindole-6-carboxylic Acid.**

Brownish-yellow cryst. from EtOH.Aq. M.p.  $313^\circ$ . Spar. sol. ord. org. solvents.

Fileti, Cairola, *Gazz. chim. ital.*, 1892, **22**, ii, 392.

**Oxindone.**

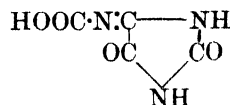
1 : 2-Diketohydrindene, *q.v.*

**Oxine.**

See 8-Hydroxyquinoline.

**Oxisatin.**

Coumarandione, *q.v.*

**Oxonic Acid (Allantoxanic acid)** $\text{C}_4\text{H}_3\text{O}_4\text{N}_3$ 

MW, 157

M.p. anhyd. about  $261^\circ$  decomp.

Biltz, Robi, *Ber.*, 1920, **53**, 1967.

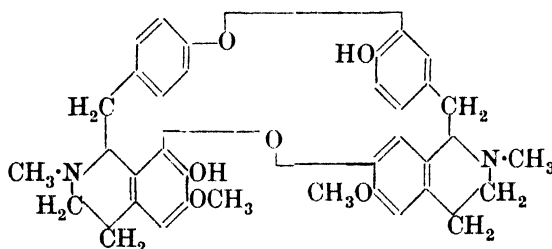
**Oxonitine** $\text{C}_{31}\text{H}_{41}\text{O}_{12}\text{N}$ 

MW, 619

Needles from AcOH- $\text{Me}_2\text{CO}$ . M.p.  $277^\circ$  decomp. Spar. sol. ord. org. solvents.  $[\alpha]_D^{25} - 48.18^\circ$  in  $\text{CHCl}_3$ .

Majima, Sugimoto, Shimanuki, *Ber.*, 1932, **65**, 595 (*Bibl.*).

Lawson, *J. Chem. Soc.*, 1936, 82.

**Oxyacanthine**

Suggested structure

 $\text{C}_{38}\text{H}_{38}\text{O}_6\text{N}_2$ 

MW, 618

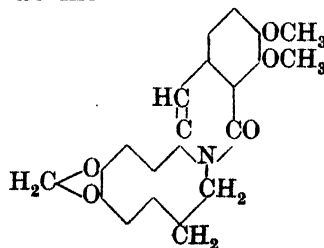
Occurs in root of *Berberis vulgaris*. Needles from EtOH. M.p.  $217^\circ$  ( $208-14^\circ$ ). Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol. ligroin.  $[\alpha]_D^{25} + 131.6^\circ$  in  $\text{CHCl}_3$ .

B,HCl: m.p.  $270^\circ$ .

B,HBr: m.p.  $273^\circ$ .

Me ether:  $\text{C}_{39}\text{H}_{40}\text{O}_6\text{N}_2$ . MW, 632. B,HCl: m.p.  $261^\circ$ .

Bruchhausen, Gericke, *Chem. Zentr.*, 1931, I, 2761.

**Oxyberberine** $\text{C}_{20}\text{H}_{17}\text{O}_5\text{N}$ 

MW, 351

Yellow needles from EtOH. M.p. 198–200°. Sol. hot AcOH. Spar. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

Perkin, Ray, Robinson, *J. Chem. Soc.*, 1925, 742.

Haworth, Koepfli, Perkin, *J. Chem. Soc.*, 1927, 552.

Späth, Quietensky, *Ber.*, 1925, 58, 2267.

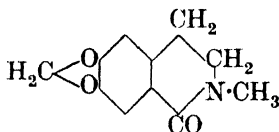
### Oxycyanogen (*Oxycyan*)

(CNO)<sub>x</sub> (CNO)<sub>x</sub> MW, (42)<sub>x</sub>

Plates. M.p. –12.5 to –11.5°. Decomp. at ord. temp. except in solution. Sol. CS<sub>2</sub>, CCl<sub>4</sub>. Liberates I from KI.

Hunt, *J. Am. Chem. Soc.*, 1932, 54, 907.

### Oxyhydrastinine



C<sub>11</sub>H<sub>11</sub>O<sub>3</sub>N MW, 205

Needles from ligroin. M.p. 97–8°. Mod. sol. EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, AcOEt, CS<sub>2</sub>. KMnO<sub>4</sub> → hydraetic acid.

B, HBr: m.p. 200°.

B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>: m.p. 160°.

Freund, *Ber.*, 1889, 22, 457.

Perkin, *J. Chem. Soc.*, 1890, 57, 1034.

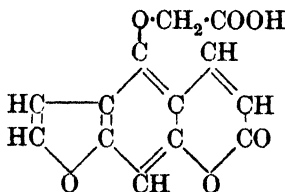
### Oxy-Koch Acid.

See 1-Naphthol-3 : 6 : 8-trisulphonic Acid.

### Oxyneurine.

See Betaine.

### Oxypeucedanic Acid



C<sub>13</sub>H<sub>8</sub>O<sub>6</sub> MW, 260

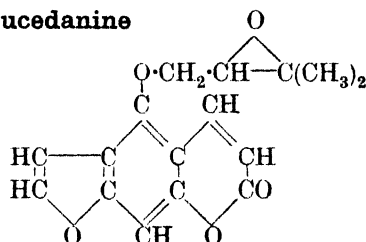
Cryst. from AcOH.Aq. M.p. 265°.

Me ester: C<sub>14</sub>H<sub>10</sub>O<sub>6</sub>. MW, 274. Cryst. from MeOH.Aq. M.p. 185°.

Späth, Klager, *Ber.*, 1933, 66, 921.

Butenandt, Marten, *Ann.*, 1932, 495, 209.

### Oxypeucedanine



C<sub>16</sub>H<sub>14</sub>O<sub>5</sub> MW, 286

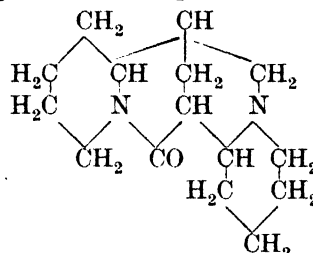
Occurs in root of *Imperatoria ostruthium*, Linn. Cryst. from Et<sub>2</sub>O-CHCl<sub>3</sub>. M.p. 142–3°. KOH fusion → phloroglucinol.

Hydrate: cryst. from AcOEt or MeOH.Aq. M.p. 134°. Monoacetyl: cryst. from Me<sub>2</sub>CO-pet. ether. M.p. 139°. Diacetyl: cryst. from CHCl<sub>3</sub>-pet. ether. M.p. 132–3° (136°). Monobenzoyl: cryst. from EtOH.Aq. M.p. 172–172.5°. Phenylurethane: cryst. from AcOEt-pet. ether. M.p. 174°.

Späth, Klager, *Ber.*, 1933, 66, 914.

Butenandt, Marten, *Ann.*, 1932, 495, 205.

### Oxysparteine (*Isolupanine*)



C<sub>15</sub>H<sub>24</sub>ON<sub>2</sub> MW, 248

Cryst. from pet. ether. M.p. 111°.

B, HI: prisms from EtOH. M.p. 275°.

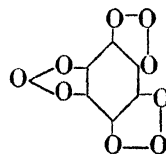
Methiodide: m.p. 203–4°.

Clemons, Morgan, Raper, *J. Chem. Soc.*, 1936, 1025.

### Oxytiglic Acid.

See 1 : 2-Dimethylglycidic Acid.

### Ozobenzene (*Benzene triozone*)



C<sub>6</sub>H<sub>6</sub>O<sub>3</sub> MW, 222

Amorp. Explodes at 50°. Very violent explosive. Insol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>, ligroin. H<sub>2</sub>O → CO<sub>2</sub> + H·COOH + CH<sub>3</sub>·COOH.

Renard, *Bull. soc. chim.*, 1895, 13, 940.

Anon., *Chem. Abstracts*, 1920, 14, 1220.



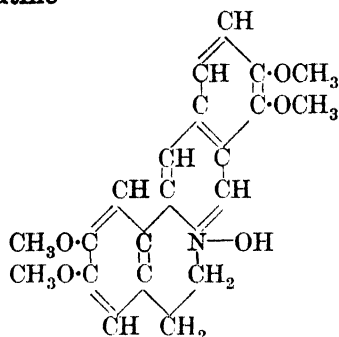
## P

**Pachycarpine.**

See Sparteine.

**Pæonol.**

See Peonol.

**Palmatine** $C_{21}H_{23}O_5N$ 

MW, 369

Occurs in East African Calumba root.

**Iodide**:  $C_{21}H_{22}O_4NI \cdot 2H_2O$ . Orange-yellow needles from  $H_2O$ . M.p.  $241^\circ$  decomp. ( $238-40^\circ$  decomp.). Mod. sol. hot  $H_2O$ , EtOH.

**B,  $HNO_3 \cdot 2H_2O$** : yellow needles. M.p.  $239^\circ$  decomp.

**Chloride**:  $C_{21}H_{22}O_4NCl \cdot 3H_2O$ . Greenish-yellow needles from  $H_2O$ . M.p.  $205^\circ$  decomp. Sol.  $H_2O$ , EtOH.

**Sulphate**:  $(C_{21}H_{22}O_4N)_2SO_4 \cdot 5H_2O$ . Yellow needles. M.p.  $250^\circ$ . Sol. EtOH. Mod. sol. cold  $H_2O$ .

**$(C_{21}H_{22}O_4NCl)_2 \cdot PtCl_4$** : yellow cryst. M.p.  $236^\circ$ .

Feist, Sandstedt, *Arch. Pharm.*, 1918, **256**, 1.

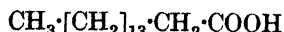
Feist, Awe, Etzrodt, *Chem. Zentr.*, 1935, I, 2374.

Späth, Quietensky, *Ber.*, 1925, **58**, 2267.

Haworth, Koepfli, Perkin, *J. Chem. Soc.*, 1927, 548.

**Palmitaldehyde.**

See Palmitic Aldehyde.

**Palmitic Acid (Hexadecylic acid)** $C_{16}H_{32}O_2$ 

MW, 256

Occurs in form of esters (glycerides) in oils and fats of vegetable and animal origin. Usually obtained from palm-oil, Japan wax or Chinese vegetable tallow. M.p.  $63-4^\circ$  ( $62-6^\circ$ ). B.p.  $390^\circ$ ,  $268.5^\circ/100$  mm.,  $219^\circ/20$  mm.,  $215^\circ/15$  mm.  $D_4^{25}$  0.8527 (liq.),  $D_4^{25}$

0.8465,  $D_4^{70}$  0.8487,  $D_4^{80}$  0.8347.  $n_D^{50}$  1.4339,  $n_D^{70}$  1.4304,  $n_D^{74.5}$  1.4284,  $n_D^{80}$  1.42691. Insol.  $H_2O$ . Sol. 11 parts EtOH at  $20^\circ$ , 100 parts 94-5% EtOH at  $0^\circ$ , 256 parts 75% EtOH at  $20^\circ$ , 2000 parts 50% EtOH at  $10^\circ$ . Sol.  $Et_2O$ . Spar. sol. pet. ether. Alk.  $KMnO_4 \rightarrow$  mixture of mono- and di-carboxylic acids of smaller carbon content. Chars with conc.  $H_2SO_4$  at  $160-80^\circ$  giving CO and  $SO_2$ .  $P_2O_5$  at  $200-10^\circ \rightarrow$  palmitone.

$C_{16}H_{31}O_2NH_4 + C_{16}H_{32}O_2$ : insol. cold  $H_2O$ .  $2(C_{16}H_{31}O_2Na) + C_{16}H_{32}O_2$ : m.p.  $115-17^\circ$ . Insol.  $H_2O$ . Sol. hot EtOH.

$C_{16}H_{31}O_2Na + C_{16}H_{32}O_2$ : m.p.  $97-8^\circ$ .

$C_{16}H_{31}O_2Na$ : cryst. from EtOH. M.p. about  $270^\circ$ .

$C_{16}H_{31}O_2Na + 2C_{16}H_{32}O_2$ : m.p.  $81-2^\circ$ .

$C_{16}H_{31}O_2K + C_{16}H_{32}O_2$ : cryst.

$C_{16}H_{31}O_2K$ : white tablets. Sol. EtOH.

$(C_{16}H_{31}O_2)_2Ca$ : spar. sol.  $H_2O$ .

**Me ester**:  $C_{17}H_{34}O_2$ . MW, 270. Cryst. from EtOH at  $-15^\circ$ . M.p.  $30.5^\circ$  ( $29.5^\circ$ ). B.p.  $415-18^\circ/747$  mm.,  $190.5^\circ$  ( $196^\circ$ )/15 mm.,  $184^\circ/12$  mm.

**Et ester**:  $C_{18}H_{36}O_2$ . MW, 284. Needles. M.p.  $25^\circ$  ( $23.5^\circ$ ). B.p.  $184.5-185.5^\circ$  ( $191^\circ$ )/10 mm.  $n_D^{50}$  1.4278,  $n_D^{70}$  1.4200.

**2-Chloroethyl ester**:  $C_{18}H_{35}O_2Cl$ . MW, 318.5. Plates from EtOH. M.p.  $41.5^\circ$  (about  $44^\circ$ ). B.p.  $138^\circ$  in high vacuum.

**2-Bromoethyl ester**:  $C_{18}H_{35}O_2Br$ . MW, 363. M.p.  $62^\circ$ . B.p.  $144^\circ$  in high vacuum.

**2-Iodoethyl ester**:  $C_{18}H_{35}O_2I$ . MW, 410. Plates from EtOH. M.p.  $54^\circ$ .

**Propyl ester**:  $C_{19}H_{38}O_2$ . MW, 298. Needles. M.p.  $20.4^\circ$  ( $18.8-19.2^\circ$ ).  $n_D^{50}$  1.4290,  $n_D^{70}$  1.4211.

**Butyl ester**:  $C_{20}H_{40}O_2$ . MW, 312. M.p.  $16.9^\circ$ .  $n_D^{50}$  1.4312,  $n_D^{70}$  1.4232.

**n-Amyl ester**:  $C_{21}H_{42}O_2$ . MW, 326. M.p.  $19.4^\circ$ .  $n_D^{50}$  1.4320,  $n_D^{70}$  1.4241.

**Isoamyl ester**: m.p.  $12.5^\circ$  ( $9^\circ$ ).  $n_D^{50}$  1.4315,  $n_D^{70}$  1.4235.

**Heptyl ester**:  $C_{23}H_{46}O_2$ . MW, 354. Liq. Solidifies at  $8-10^\circ$ .

**Octyl ester**:  $C_{24}H_{48}O_2$ . MW, 368. M.p.  $22.5^\circ$ .  $n_D^{50}$  1.4358,  $n_D^{70}$  1.4277.

**Decyl ester**:  $C_{26}H_{52}O_2$ . MW, 396. M.p.  $30^\circ$ .

**Dodecyl ester**:  $C_{28}H_{56}O_2$ . MW, 424. Plates from EtOH. M.p.  $41^\circ$ .

**Tetradecyl ester**:  $C_{30}H_{60}O_2$ . MW, 452. M.p.  $48^\circ$ .

**Pentadecyl ester**:  $C_{31}H_{62}O_2$ . MW, 466. M.p.  $57^\circ$  ( $55.5^\circ$ ).

*Hexadecyl (cetyl) ester*:  $C_{32}H_{64}O_2$ . MW, 480. Chief constituent of spermaceti. Plates from  $Et_2O$ . M.p. 53–4° (51–6°).

*Octadecyl ester*:  $C_{34}H_{68}O_2$ . MW, 508. M.p. 59°.

*Ceryl ester*:  $C_{42}H_{84}O_2$ . MW, 620. Main constituent of opium wax. M.p. 79°.

*Melissyl ester*:  $C_{46}H_{92}O_2$ . MW, 676. A constituent of beeswax. M.p. 72°.

*Vinyl ester*:  $C_{19}H_{34}O_2$ . MW, 294. B.p. 165°/2 mm.

*Allyl ester*:  $C_{19}H_{34}O_2$ . MW, 294. M.p. 20–5°.

*Ethylene glycol mono-ester*:  $C_{18}H_{36}O_3$ . MW, 300. M.p. 51·5°.

*Ethylene glycol di-ester*:  $C_{34}H_{66}O_4$ . MW, 538. M.p. 72° (69°). B.p. 226° in high vacuum.

*Glycerol mono-ester*: see Monopalmitin.

*Glycerol di-ester*: see Dipalmitin.

*Glycerol tri-ester*: see Tripalmitin.

*Phenyl ester*:  $C_{22}H_{36}O_2$ . MW, 332. M.p. 45°. B.p. 249·5°/15 mm.

*o-Nitrophenyl ester*:  $C_{22}H_{35}O_4N$ . MW, 377. Needles from  $EtOH$ . M.p. 51–2°.

*p-Tolyl ester*:  $C_{23}H_{38}O_2$ . MW, 346. M.p. 47°. B.p. 258°/15 mm.

*Benzyl ester*:  $C_{23}H_{38}O_2$ . MW, 346. M.p. 36°.

*p-Nitrobenzyl ester*:  $C_{23}H_{37}O_4N$ . MW, 391. M.p. 42–42·5°.

*Phenacyl ester*:  $C_{24}H_{38}O_3$ . MW, 374. M.p. 63°.

*p-Chlorophenacyl ester*:  $C_{24}H_{37}O_3Cl$ . MW, 408·5. M.p. 82°.

*p-Bromophenacyl ester*:  $C_{24}H_{37}O_3Br$ . MW, 454. M.p. 86° (81·5°).

*p-Iodophenacyl ester*:  $C_{24}H_{37}O_3I$ . MW, 500. M.p. 90°.

*p-Nitrophenacyl ester*:  $C_{24}H_{37}O_5N$ . MW, 419. M.p. 42·5°.

*Amide*:  $C_{16}H_{33}ON$ . MW, 255. M.p. 106–7°. B.p. 235–6°/12 mm.

*Anilide*: needles from  $EtOH$ . M.p. 90·5° (87·5°). B.p. 282–4°/17 mm., 132·5°/10 mm. Sol.  $EtOH$ ,  $Me_2CO$ , warm  $AcOH$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. pet. ether. Insol.  $H_2O$ .

*p-Bromoanilide*: m.p. 110°.

*2:4:6-Tribromoanilide*: m.p. 124°.

*p-Toluidide*: cryst. from  $EtOH$ . M.p. 96° (93·5°). B.p. 140°/10 mm.

*1-Naphthylamide*: needles. M.p. 112·8° (106°). B.p. 182°/10 mm.

*2-Naphthylamide*: cryst. from  $EtOH$ . M.p. 109°. B.p. 198·5°/10 mm.

*Phenyldiazide*: m.p. 110·5°.

*Diphenylhydrazide*: m.p. 124°.

*2-Naphthylhydrazide*: m.p. 135°.

*Anhydride*:  $C_{32}H_{62}O_3$ . MW, 494. M.p. 64°.  $D_4^{20}$  0·847,  $D_4^{25}$  0·8383.  $n_D^{25}$  1·4364,  $n_D^{100}$  1·4679.

*Chloride*:  $C_{16}H_{31}OCl$ . MW, 274·5. M.p. 12°. B.p. 194–5°/17 mm., 198–200°/15 mm.

*Nitrile*:  $C_{16}H_{31}N$ . MW, 237. M.p. 31° (29°). B.p. 251°/100 mm., 196°/15 mm.  $D_4^{25}$  0·8224 (liq.),  $D_4^{20}$  0·8186.

Krafft, *Ber.*, 1888, **21**, 2265.

Chittenden, Smith, *Am. Chem. J.*, 1884, **6**, 218.

Claus, v. Dreden, *J. prakt. Chem.*, 1891, **43**, 149.

Klages, *Ber.*, 1902, **35**, 2260.

Kreis, Hafner, *Ber.*, 1903, **36**, 2769.

Charitschkow, *Chem. Zentr.*, 1905, **II**, 118.

Büchel, D.R.P., 281,364, (*Chem. Zentr.*, 1915, **I**, 230).

Holde, Ripper, Zadek, *Ber.*, 1924, **57**, 103.

Whitby, *J. Chem. Soc.*, 1926, 1462.

Wilkie, *J. Soc. Chem. Ind.*, 1927, **46**, 471T.

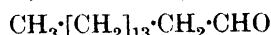
Hann, Reid, Jamieson, *J. Am. Chem. Soc.*, 1930, **52**, 818.

Dubovitz, *Chem.-Ztg.*, 1930, **54**, 814.

Smith, *J. Chem. Soc.*, 1931, 802.

Deutsche Hydrierwerke, A-G., D.R.P., 572,867, (*Chem. Abstracts*, 1933, **27**, 4433).

**Palmitic Aldehyde** (*Palmitaldehyde*, *hexadecylic aldehyde*, *hexadecanal*)



$C_{16}H_{32}O$  MW, 240

M.p. 34°. B.p. 200–2°/29 mm. Sol. most org. solvents. Insol.  $H_2O$ . Polymerises on keeping to trimeride (needles from  $Et_2O$ , m.p. 73°).

*Oxime*: needles from dil.  $EtOH$ . M.p. 88°. Sol.  $Et_2O$ ,  $CHCl_3$ . Spar. sol. pet. ether,  $C_6H_6$ .

*Semicarbazone*: plates from dil.  $EtOH$ . M.p. 109° (107°). Sol. hot  $CHCl_3$ ,  $C_6H_6$ .

*Thiosemicarbazone*: m.p. 109°.

*p-Nitrophenylhydrazone*: yellow cryst. M.p. 96·5°.

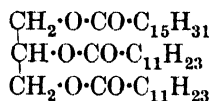
Stephen, *J. Chem. Soc.*, 1925, 1876.

Feulgen, Behrens, *Z. physiol. Chem.*, 1928, **177**, 229

Gottfried, Ulzer, *Chem. Abstracts*, 1929, **23**, 1902,

Le Sueur, *J. Chem. Soc.*, 1905, **87**, 1892.

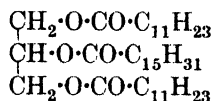
Krafft, *Ber.*, 1880, **13**, 1416.

$\alpha$ -Palmito- $\alpha'$ - $\beta$ -dilaurin
 $\text{C}_{43}\text{H}_{82}\text{O}_6$  MW, 694

Cryst. from EtOH. M.p. 47-8° (44-8°).  $n_D^{70}$  1.43965. 100 ccs. EtOH dissolve 0.54 gm. at 23°. Sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ ,  $\text{Et}_2\text{O}$ .

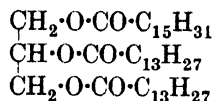
Fischer, Bergmann, Bärwind, *Ber.*, 1920, 53, 1605.

McElroy, King, *J. Am. Chem. Soc.*, 1934, 56, 1192.

 $\beta$ -Palmito- $\alpha\alpha'$ -dilaurin
 $\text{C}_{43}\text{H}_{82}\text{O}_6$  MW, 694

M.p. 47-8°.  $n_D^{70}$  1.43980. 100 ccs. EtOH dissolve 0.38 gm. at 23°.

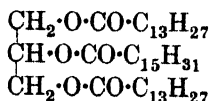
See second reference above.

 $\alpha$ -Palmito- $\alpha'$ - $\beta$ -dimyristin
 $\text{C}_{47}\text{H}_{90}\text{O}_6$  MW, 750

M.p. 53.0° (47.8°). Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , hot EtOH. 100 ccs. EtOH dissolve 0.42 gm. at 27.5°. 100 ccs.  $\text{Me}_2\text{CO}$  dissolve 1.82 gm. at 27.5°.

Averill, Roche, King, *J. Am. Chem. Soc.*, 1929, 51, 870.

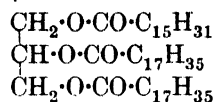
Heiduschka, Schuster, *J. prakt. Chem.*, 1928, 120, 153.

 $\beta$ -Palmito- $\alpha\alpha'$ -dimyristin
 $\text{C}_{47}\text{H}_{90}\text{O}_6$  MW, 750

M.p. 59.8-60° (49.5°). Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , hot EtOH. 100 ccs. EtOH dissolve 0.10 gm. at 27.5°. 100 ccs.  $\text{Me}_2\text{CO}$  dissolve 0.61 gm. at 27.5°.

Heiduschka, Schuster, *J. prakt. Chem.*, 1928, 120, 152.

See also first reference above.

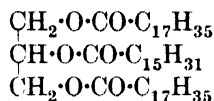
 $\alpha$ -Palmito- $\alpha'$ - $\beta$ -distearin
 $\text{C}_{55}\text{H}_{106}\text{O}_6$  MW, 862

Occurs in lard. Plates from  $\text{C}_6\text{H}_6$ -EtOH. M.p. 68.5° (63.2°, 62.6°).  $n_D^{70}$  1.44245.

Amberger, Bromig, *Biochem. Z.*, 1922, 130, 261.

Robinson, Roche, King, *J. Am. Chem. Soc.*, 1932, 54, 708.

Bömer, *Chem. Zentr.*, 1913, I, 1620.

 $\beta$ -Palmito- $\alpha\alpha'$ -distearin
 $\text{C}_{55}\text{H}_{106}\text{O}_6$  MW, 862

Occurs in lard and in the fat of beef and mutton. Cryst. from  $\text{Et}_2\text{O}$ . M.p. 63.3° (67.9°, 64.8°, 68.0°).  $n_D^{70}$  1.4467 (1.44374).

Kreis, Hafner, *Ber.*, 1903, 36, 1124, 2766.

Bömer, Limprich, *Chem. Zentr.*, 1913, I, 1621.

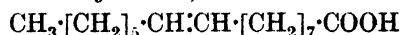
Schuster, *J. pharm. chim.*, 1932, 16, 421.

Amberger, Bromig, *Biochem. Z.*, 1922, 130, 262.

Whitby, *J. Chem. Soc.*, 1926, 1461.

Robinson, Roche, King, *J. Am. Chem. Soc.*, 1932, 54, 708.

**Palmitoleic Acid** (Zoomaric acid, 8-hexadecenoic acid,  $\Delta^8$ -hexadecylenic acid, 8-pentadecylene-1-carboxylic acid)


 $\text{C}_{16}\text{H}_{30}\text{O}_2$  MW, 254

Found as glyceride in oil of whale, cod, walrus, seal, etc. Liq. Dil. alk.  $\text{KMnO}_4$  in cold  $\rightarrow$  8:9-dihydroxypalmitic acid.

*Me ester*:  $\text{C}_{17}\text{H}_{32}\text{O}_2$ . MW, 268. B.p. 140-1°/5 mm.  $\text{H} \rightarrow$  methyl palmitate.  $\text{KMnO}_4 \rightarrow$  heptylic and azelaic acids.

*Et ester*:  $\text{C}_{18}\text{H}_{34}\text{O}_2$ . MW, 282. Liq.

Armstrong, Hilditch, *J. Soc. Chem. Ind.*, 1925, 44, 182T. (See also Hilditch, Vidyarthi, *J. Soc. Chem. Ind.*, 1927, 46, 172T.)

Schmidt-Nielsen, *Chem. Zentr.*, 1922, I, 1047.

Toyama, *J. Soc. Chem. Ind. Japan*, 1927, 30, 603.

**Palmitone** (*Di-pentadecyl ketone, hentriacontanone-16, 16-ketohentriacontane*)



$\text{C}_{31}\text{H}_{62}\text{O}$

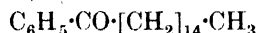
MW, 450

Leaflets from EtOH. M.p.  $82.8^\circ$ .  $D_4^{20}$  0.7947. Does not form bisulphite comp.

*Oxime*: needles from EtOH or AcOH. M.p.  $59^\circ$ .

Kipping, *J. Chem. Soc.*, 1890, **57**, 985.

**Palmitophenone** (*Pentadecyl phenyl ketone, palmitylbenzene*)



$\text{C}_{22}\text{H}_{36}\text{O}$

MW, 316

Plates from EtOH. M.p.  $59^\circ$ . B.p.  $250.5-251^\circ/15$  mm. Mod. sol.  $\text{Et}_2\text{O}$ . Prac. insol. cold EtOH.  $D_4^{20}$  0.8692.  $n_D^{20}$  1.46746. Triboluminescent.  $\text{CrO}_3 \rightarrow$  benzoic and pentadecylic acids.

Krafft, *Ber.*, 1886 **19**, 2982.

**Palmitylacetic Acid.**

See 2-Ketostearic Acid.

**p-Palmitylanisole.**

See under p-Hydroxypalmitophenone.

**Palmitylglycine**



$\text{C}_{18}\text{H}_{35}\text{O}_3\text{N}$

MW, 313

Cryst. from  $\text{Me}_2\text{CO}$  or EtOH. M.p.  $125^\circ$  ( $121^\circ$ ). Sinters at  $119^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ , hot  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ , pet. ether.

*Et ester*:  $\text{C}_{20}\text{H}_{39}\text{O}_3\text{N}$ . MW, 341. Needles from  $\text{Et}_2\text{O}$ . M.p.  $80^\circ$ .

Bondi, Frankl, *Biochem. Z.*, 1909, **17**, 552.

Abderhalden, Funk, *Z. physiol. Chem.*, 1910, **65**, 62.

Karrer, Miyamichi, Storm, Widmer, *Helv. Chim. Acta.*, 1925, **8**, 205.

**p-Palmitylphenetole.**

See under p-Hydroxypalmitophenone.

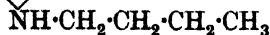
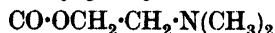
**p-Palmitylphenol.**

See p-Hydroxypalmitophenone.

**Panax sapogenin.**

See Oleanolic Acid.

**Pantocaine** (*N-Butylnovocaine, 2-dimethyl-aminoethyl ester of p-butylaminobenzoic acid*)



$\text{C}_{15}\text{H}_{24}\text{O}_2\text{N}_2$

MW, 264

M.p.  $153-4^\circ$ . Local anæsthetic. Fifteen times as effective as novocaine. Nearly ten times as active as cocaine, but more toxic. Said to cause irritation and turbidity of the cornea.

$B, \text{HCl}$ : m.p.  $147-8^\circ$ . Sol.  $\text{H}_2\text{O}$ .

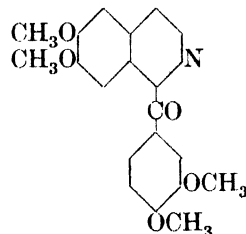
$B, \text{HNO}_3$ : m.p.  $131-2^\circ$ .

Fussgänger, Schaumann, *Chem. Abstracts*, 1932, **26**, 515.

Eisler, U.S.P., 1,889,645, (*Chem. Abstracts*, 1933, **27**, 1717).

I.G., D.R.P., 582,715, (*Chem. Abstracts*, 1934, **28**, 778).

**Papaveraldine** (6 : 7-Dimethoxy-1-veratroyl-isoquinoline, xanthaline)



$\text{C}_{20}\text{H}_{19}\text{O}_5\text{N}$

MW, 353

Occurs in opium. Cryst. from  $\text{C}_6\text{H}_6$  or pet. ether. M.p.  $210^\circ$  ( $208^\circ$ ). Sol. min. acids, hot AcOH. Mod. sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol. EtOH,  $\text{Et}_2\text{O}$ , pet. ether. Insol.  $\text{H}_2\text{O}$ .  $\text{Zn} + \text{H}_2\text{SO}_4$  or AcOH  $\rightarrow$  papaverinol.  $\text{Zn} + \text{Ac}_2\text{O} \rightarrow$  papaverine.  $\text{HNO}_3 \rightarrow$  nitropapaveraldine. KOH fusion  $\rightarrow$  veratric acid + 6 : 7-dimethoxyisoquinoline.

$B, \text{HCl}, 2\frac{1}{2} (?) \text{H}_2\text{O}$ : yellow cryst. from  $\text{H}_2\text{O}$ .

$B, \text{H}_2\text{SO}_4$ : yellow needles. Decomp. by  $\text{H}_2\text{O}$ .

$B, \text{HNO}_3, 2\text{H}_2\text{O}$ : yellow needles. Decomp. by hot  $\text{H}_2\text{O}$ .

$B, \text{H}_2\text{PtCl}_6, \text{H}_2\text{O}$ : orange-red prisms from dil. HCl. Decomp. at  $210^\circ$ .

*Picrate*: yellow needles. M.p.  $208-9^\circ$ .

*Methiodide*,  $\text{H}_2\text{O}$ : yellow prisms. M.p.  $194^\circ$ .

*Methiodide*,  $3\text{H}_2\text{O}$ : yellow prisms. M.p.  $136^\circ$  ( $132^\circ$ ).

*Oxime*: exists in two forms. (i) Needles or prisms. M.p.  $235-6^\circ$ ; (ii) prisms from EtOH. M.p.  $245^\circ$ .

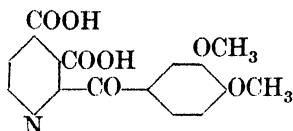
*Phenylhydrazone*: reddish-yellow aggregates from dil. EtOH. M.p.  $80-1^\circ$ .

Stuchlik, *Monatsh.*, 1900, **21**, 828.

Pschorr, *Ber.*, 1904, **37**, 1936.

Dobson, Perkin, *J. Chem. Soc.*, 1911, **99**, 135.

Buck, Haworth, Perkin, *J. Chem. Soc.*, 1924, **125**, 2184.

**Papaveric Acid** (2-Veratroylcinchomeronic acid) $C_{16}H_{13}O_7N$ 

MW, 331

Cryst. +  $H_2O$  from  $H_2O$ . M.p.  $233^\circ$  decomp. Sol. hot dil. EtOH, AcOH, dil. HCl. Spar. sol. cold  $H_2O$ , EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ , pet. ether. Sol. conc.  $H_2SO_4$  with reddish-yellow col. KOH fusion  $\rightarrow$  protocatechuic acid.  $Ac_2O \rightarrow$  papaveric anhydride.  $HNO_3 \rightarrow$  nitropapaveric acid.

$K_2A, 2\frac{1}{2}H_2O$ : plates from dil. EtOH. Sol.  $H_2O$ . Spar. sol. EtOH.

$B, HCl$ : orange-red cryst. from HCl.Aq.

$B, HCl, 2\frac{1}{2}H_2O$ : yellow needles.

Oxime: needles from EtOH. M.p.  $154-7^\circ$ . Sol.  $H_2O$ , EtOH.

Phenylhydrazone: yellow needles from EtOH. M.p.  $190^\circ$ .

3-Me ester:  $C_{17}H_{13}O_7N$ . MW, 345. Cryst. from MeOH. M.p.  $156^\circ$ . Sol. MeOH. Spar. sol.  $H_2O$ .

4-Me ester: needles from  $H_2O$  or MeOH. M.p.  $198^\circ$  decomp. ( $196^\circ$ ). Sol. EtOH,  $Me_2CO$ , AcOEt. Spar. sol.  $H_2O$ , MeOH. Insol.  $Et_2O$ .

Di-Me ester:  $C_{18}H_{17}O_7N$ . MW, 359. Plates from  $Me_2CO$ . M.p.  $121-2^\circ$  ( $122-4^\circ$ ). Sol.  $Me_2CO$ , AcOEt.

3-Et ester:  $C_{18}H_{17}O_7N$ . MW, 359. Needles. M.p.  $187-8^\circ$ . Spar. sol.  $H_2O$ .

4-Et ester: needles from EtOH. M.p.  $184^\circ$ . Spar. sol.  $H_2O$ .

Anhydride:  $C_{32}H_{24}O_{13}N_2$ . MW, 644. Needles from  $C_6H_6$ . M.p.  $169-70^\circ$ .

Methochloride,  $H_2O$ : yellow plates. M.p.  $182-4^\circ$  decomp.

Goldschmiedt, *Monatsh.*, 1885, 6, 380; 1888, 9, 357.

Goldschmiedt, Strache, *Monatsh.*, 1889, 10, 159, 692.

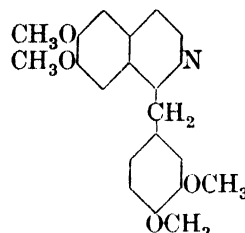
Goldschmiedt, Kirpal, *Monatsh.*, 1896, 17, 496.

Kirpal, *Monatsh.*, 1897, 18, 464.

Schranzhofer, *Monatsh.*, 1893, 14, 525.

Wegscheider, *Monatsh.*, 1902, 23, 338, 388.

Goldschmiedt, Honigschmid, *Monatsh.*, 1903, 24, 681.

**Papaverine** (6:7-Dimethoxy-1-veratrylisoquinoline, papaveroline tetramethyl ether) $C_{20}H_{21}O_4N$ 

MW, 339

Occurs in opium (0.8-1.0%). Prisms from EtOH- $Et_2O$ . Needles from  $CHCl_3$ -pet. ether. M.p.  $147^\circ$ . Sol. hot EtOH,  $CHCl_3$ ,  $Me_2CO$ . Mod. sol. hot  $C_6H_6$ . Prac. insol.  $H_2O$ , pet. ether. 1 part dissolves in 86 parts 97% EtOH at  $15^\circ$  and in 258 parts  $Et_2O$  at  $10^\circ$ . Sol. conc.  $H_2SO_4$  colourless, changing to violet on warming.  $D_4$  1.308-1.337. Optically inactive. Triboluminescent.  $Na_2Cr_2O_7 \rightarrow$  papaveraldine.  $KMnO_4 \rightarrow$  papaveraldine, papaveric acid, 6:7-dimethoxyisoquinoline-1-carboxylic acid, pyridine-2:3:4-tricarboxylic acid, meta-hemipinic acid, veratric acid, oxalic acid,  $CO_2$  and  $NH_3$ .  $Sn + HCl \rightarrow$  tetrahydropapaverine.  $Br \rightarrow$  bromopapaverine.  $HNO_3 \rightarrow$  nitropapaverine. Nitrous fumes  $\rightarrow$  nitroso-papaverine.  $HNO_2 \rightarrow$  papaveraldoxime, m.p.  $235-6^\circ$ . Boiling conc. HCl  $\rightarrow$  papaveroline dimethyl ether. HI  $\rightarrow$  papaveroline. Dist. with KOH  $\rightarrow$  methylamine, protocatechuic acid, homocatechol dimethyl ether and oxalic acid.

$B, HCl$ : plates from  $H_2O$ . M.p.  $220-1^\circ$  decomp. ( $210-13^\circ$  decomp.). Sol. 37.3 parts  $H_2O$  at  $18^\circ$ .

$B, HBr$ : cryst. from  $H_2O$  or dil. EtOH. M.p.  $213-14^\circ$  decomp.

$B, HI$ : m.p.  $200^\circ$  decomp. Sol. hot  $H_2O$ , EtOH. Dimorphous.

$B, HFeCl_4, H_2O$ : red plates. M.p.  $195^\circ$ .

$B, HFeCl_4, 2H_2O$ : orange prisms. M.p.  $195^\circ$ .

$B_2, H_2PtCl_6, \frac{1}{2}H_2O$ : m.p.  $186^\circ$ .

$B_2, H_2PtCl_6, 2H_2O$ : orange prisms from conc. HCl. M.p.  $198^\circ$ . Insol.  $H_2O$ , EtOH,  $Et_2O$ .

Acid oxalate: needles or prisms from  $H_2O$ . M.p.  $196^\circ$ . Sol. hot  $H_2O$ . Spar. sol. hot EtOH.

Succinate: plates from EtOH. M.p.  $171^\circ$ . Sol. hot  $H_2O$ .

Salicylate: plates from EtOH. M.p.  $130^\circ$ .

Benzoate: cryst. from EtOH. M.p.  $145^\circ$ . Sol. EtOH. Insol.  $H_2O$ .

Methiodide,  $4H_2O$ : cryst. from dil. EtOH.

M.p. 60–5° (55–60°), 195° anhyd. Sol.  $\text{CHCl}_3$ . Mod. sol.  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ .

*Ethiodide*: yellow cryst. from dil. EtOH. M.p. 216° decomp. Sol.  $\text{CHCl}_3$ . Spar. sol.  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ .

*Picrate*: yellow plates from EtOH. M.p. 183° (179° decomp., 154°).

*Picrolonate*: needles from EtOH. M.p. 221°.

Pictet, Gams, *Ber.*, 1909, **42**, 2943.

Buck, Haworth, Perkin, *J. Chem. Soc.*, 1924, **125**, 2179.

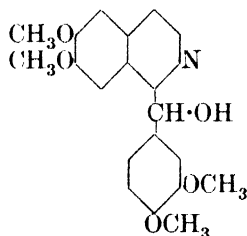
Rosenmund, Nothnagel, Riesenfeldt, *Ber.*, 1927, **60**, 392.

Späth, Burger, *ibid.*, 704.

Mannich, Walther, *Arch. Pharm.*, 1927, **265**, 1.

Kindler, Peschke, *Arch. Pharm.*, 1934, **272**, 236.

### Papaverinol (*Hydroxyxanthaline*)



$\text{C}_{20}\text{H}_{21}\text{O}_5\text{N}$

MW, 355

Prisms from dil. MeOH. M.p. 137°. Sol. MeOH, EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Ox.  $\rightarrow$  papaveraldine.

*B.HCl* yellow needles from EtOH– $\text{Et}_2\text{O}$ . M.p. 200–2°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-yellow needles from dil. HCl. M.p. 168° decomp.

*O-Benzoyl*: amorph. Sol. EtOH,  $\text{CHCl}_3$ , AcOH. *Picrate*: yellow needles from EtOH. M.p. 126° decomp.

*O-p-Bromobenzoyl*: cryst. M.p. 194°.

*Methochloride*: prisms from EtOH. M.p. 205–6°.

*Methiodide*: needles from MeOH or EtOH– $\text{Et}_2\text{O}$ . M.p. 188° (not sharp), 200° decomp. with rapid heating.

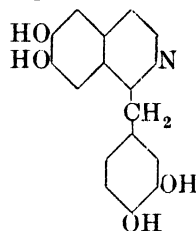
*Picrate*: yellow prisms from EtOH. M.p. 168–71° decomp.

Stuchlik, *Monatsh.*, 1900, **21**, 814.

Gadamer, *Arch. Pharm.*, 1915, **253**, 284.

King, L'Ecuyer, Pyman, *J. Chem. Soc.*, 1936, 732.

### Papaveroline (6 : 7-Dihydroxy-1-[3 : 4-dihydroxybenzyl]-isoquinoline)



$\text{C}_{16}\text{H}_{13}\text{O}_4\text{N}$

MW, 273

Cryst. powder +  $2\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  at 100°. Darkens at 150° and decomp. on further heating without melting. Sol. AcOH, glycerol. Mod. sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Insol.  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether. Sol. dil. min. acids. Dist. with Zn dust  $\rightarrow$  1-methylisoquinoline.

*B.HCl, H<sub>2</sub>O*: needles. Sol. hot  $\text{H}_2\text{O}$ .

*B.HI, 2H<sub>2</sub>O*: needles from  $\text{H}_2\text{O}$ . M.p. anhyd. 230° decomp. Mod. sol. hot  $\text{H}_2\text{O}$ .

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>, 10H<sub>2</sub>O*: loses  $\text{H}_2\text{O}$  at 105°.

*B<sub>2</sub>(COOH)<sub>2</sub>, 3H<sub>2</sub>O*: needles. Sol. hot  $\text{H}_2\text{O}$ .

*Methochloride*: m.p. 264° decomp. (235°).

*Ethochloride*: cryst. M.p. 215°.

*Benzylchloride*: m.p. 158°.

*Methiodide*: m.p. 208° decomp. From dil. HCl m.p. 77° (probably hydrated).

*Methopicrate*: m.p. 210° decomp.

*Tetra-Me ether*: see Papaverine.

*O-Tetrabenzoyl*: m.p. 148°.

Goldschmiedt, *Monatsh.*, 1885, **6**, 967.

Krauss, *Monatsh.*, 1890, **11**, 350.

Claus, Kassner, *J. prakt. Chem.*, 1897, **56**, 344.

Kitasato, Robinson, *J. Chem. Soc.*, 1932, 785.

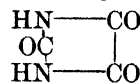
### Para-acetaldehyde.

See Paraldehyde.

### Para-anthracene.

See Dianthracene.

### Parabanic Acid (*Oxalylurea*)



$\text{C}_3\text{H}_2\text{O}_3\text{N}_2$

MW, 114

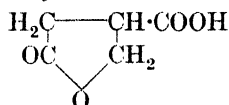
Needles or prisms from boiling  $\text{H}_2\text{O}$ . Part. sublimes at 100°. Decomp. at 243°. Sol. 21 parts  $\text{H}_2\text{O}$  at 8°. Decomp. by boiling aq. alkalis. Salts unstable.

*C<sub>3</sub>H<sub>2</sub>O<sub>3</sub>N<sub>2</sub>, H<sub>2</sub>O*: cryst. Sol. 7.4 parts  $\text{H}_2\text{O}$  at 8°. Loses  $\text{H}_2\text{O}$  at 150–60°.

Behrend, Asche, *Ann.*, 1918, **416**, 226.

Biltz, Schiemann, *Ber.*, 1926, **59**, 721.

**Paraconic Acid** (5-Ketotetrahydrofuran-3-carboxylic acid, butyrolactone- $\beta$ -carboxylic acid, 5-ketotetrahydro- $\beta$ -furoic acid)



$\text{C}_5\text{H}_6\text{O}_4$

MW, 150

Cryst. M.p.  $57-8^\circ$  ( $55^\circ$ ). Dist.  $\rightarrow$  citraconic anhydride.

Fittig, Beer, *Ann.*, 1883, **216**, 84.

Reitter, *Ber.*, 1898, **31**, 2724.

**Paracoumarone.**

See Coumarone.

**Paraformaldehyde.**

See under Formaldehyde.

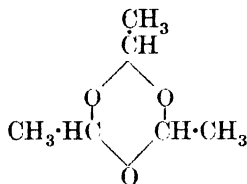
**Para-indene.**

See Indene.

**Paralactic Acid.**

See Lactic Acid.

**Paraldehyde** (Para-acetaldehyde, 2 : 4 : 6-trimethyl-1 : 3 : 5-trioxan)



$\text{C}_6\text{H}_{12}\text{O}_3$

MW, 132.

M.p.  $12-6^\circ$  ( $10^\circ$ ). B.p.  $128^\circ$  ( $124^\circ$ ). Spar. sol.  $\text{H}_2\text{O}$ .  $D_4^{10}$  1.0037,  $D_4^{15}$  0.9975,  $D_4^{20}$  0.9943,  $D_4^{25}$  0.9772,  $D_4^{30}$  0.9536,  $D_4^{35}$  0.9264,  $D_4^{40}$  0.8738.  $n_D^{20}$  1.4049. Heat of comb.  $C_p = 813.2$  Cal. Gives acetaldehyde on warming with a few drops of conc.  $\text{H}_2\text{SO}_4$ , or on boiling with dil.  $\text{HCl}$ .  $\text{Ac}_2\text{O} + \text{conc. H}_2\text{SO}_4 \rightarrow$  ethylidene diacetate.  $\text{PCl}_5 \rightarrow$  ethylidene chloride. Br water  $\rightarrow$  acetic acid. Br in  $\text{CHCl}_3 \rightarrow$  bromo- and dibromo-acetaldehyde.

Friedel, *Bull. soc. chim.*, 1893, **9**, 385.

Geuther, Cartmell, *Ann.*, 1859, **112**, 16.

Vogt, Nieuwland, *J. Am. Chem. Soc.*, 1921, **43**, 207B.

Morton, Nicolet, U.S.P., 1,300,451, (*Chem. Abstracts*, 1919, **13**, 1861).

**Paraldol** (Dimeride of aldol)

$\text{C}_8\text{H}_{16}\text{O}_4$

MW, 176

Prisms. M.p.  $90^\circ$  ( $82^\circ$ ,  $80-90^\circ$ ). B.p.  $90-100^\circ$  in high vacuo. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Less sol.  $\text{Et}_2\text{O}$ . Vac. dist.  $\rightarrow$  aldol. Ox.  $\rightarrow$  2-hydroxybutyric acid. Heat in presence of

$\text{I} \rightarrow$  crotonaldehyde.  $\text{H}(+\text{Ni}) \rightarrow \beta$ -butylene glycol.

Nowak, *Monatsh.*, 1901, **22**, 1142.

Wurtz, *Compt. rend.*, 1883, **97**, 1525.

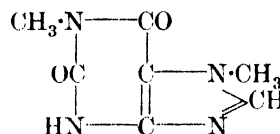
**Paranthrene.**

See Dianthracene.

**Paratophan.**

See 6-Methyl-2-phenylquinoline-4-carboxylic Acid.

**Paraxanthine** (1 : 7-Dimethylxanthine, 1 : 7-dimethyl-2 : 6-dihydroxy purine)



$\text{C}_7\text{H}_8\text{O}_2\text{N}_4$

MW, 180

Present in human urine. Plates from  $\text{H}_2\text{O}$ . M.p.  $298-9^\circ$  ( $289^\circ$ ,  $284^\circ$ ). Sol.  $\text{NH}_3$ . Aq.,  $\text{HCl} + \text{HNO}_3$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Mod. sol. hot  $\text{H}_2\text{O}$ . Insol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CH}_3\text{I} \rightarrow$  caffeine.

$\text{B.HCl.H}_2\text{O}$ : dissociates at  $100^\circ$ . Decomp. in  $\text{H}_2\text{O}$ .

$\text{B}_2\text{H}_2\text{PtCl}_6.\text{H}_2\text{O}$ : orange cryst.

$\text{B}_2\text{HAuCl}_4.\frac{1}{2}\text{H}_2\text{O}$ : m.p.  $227-8^\circ$ .

Picrate: yellow ppt. Decomp. in  $\text{H}_2\text{O}$ .

Fischer, *Ber.*, 1897, **30**, 554, 2400.

Fischer, Clemm, *Ber.*, 1898, **31**, 2623.

Fischer, Ach, *Ber.*, 1906, **39**, 423.

Traube, Dudley, *Ber.*, 1913, **46**, 3839.

Boehringer, Sohn, D.R.P., 582,435,

(*Chem. Abstracts*, 1933, **27**, 5754);

D.R.P., 576,604, (*Chem. Abstracts*, 1933, **27**, 5757).

**Parietin.**

See Physcione.

**Parigenin.**

See Sarsasapogenin.

**Patchoulene.**

See Patschulene.

**Patschulene** (Patchoulene)

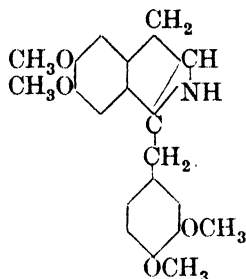
$\text{C}_{15}\text{H}_{24}$

MW, 204

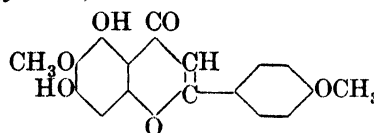
Liq. with cedarwood odour. B.p.  $255-6^\circ$ ,  $252-3^\circ/743$  mm.,  $112-15^\circ/12-12.5$  mm.  $D_4^{20}$  0.9296.  $n_D^{20}$  1.49853.  $[\alpha]_D^{20} - 38.08^\circ$ . Very sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{EtOH}$ ,  $\text{AcOH}$ . Insol.  $\text{HCl}$ ,  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ , but gives red col. with these acids.

Wallach, Tuttle, *Ann.*, 1894, **279**, 394.

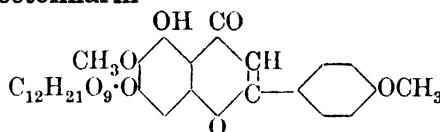
Schimmel, *Chem. Zentr.*, 1904, **I**, 1265.

**Pavine** (2 : 4-Dihydropapaverine) $C_{20}H_{23}O_4N$ 

MW, 341

*d*-.  
Cryst. from  $C_6H_6$ . M.p.  $224^\circ$ .  $[\alpha]_D^{25} + 150.3^\circ$  in  $CHCl_3$ ,  $+ 198.8^\circ$  in  $AcOH$ .*d*-Tartrate: cryst. from  $Me_2CO-Et_2O$ . M.p.  $156-8^\circ$  decomp. Very sol.  $H_2O$ , org. solvents.  $[\alpha]_D^{22} + 157.5^\circ$ .*l*-.  
Cryst. from  $C_6H_6$ . M.p.  $224^\circ$ .  $[\alpha]_D^{25} - 150.8^\circ$  in  $CHCl_3$ .*d*-Tartrate: cryst. from  $Me_2CO-Et_2O$ . M.p.  $156-8^\circ$  decomp. Very sol.  $H_2O$ , org. solvents.  $[\alpha]_D^{22} - 150.5^\circ$  in  $H_2O$ .*dl*-.  
Prisms from xylene, needles from  $EtOH$ . M.p.  $201-2^\circ$ . Sol.  $CHCl_3$ ,  $CS_2$ , warm  $Me_2CO$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ , pet. ether.*B*, *HCl*: cryst.  $+ 5H_2O$ . Decomp. at  $325^\circ$ .  
*N*-Me:  $C_{21}H_{25}O_4N$ . MW, 355. Prisms from  $Et_2O$  or  $C_6H_6$ . M.p.  $140-1^\circ$ . Sol. most org. solvents. Spar. sol.  $H_2O$ ,  $Et_2O$ , pet. ether.Conc.  $H_2SO_4 \rightarrow$  weak yellowish-green col.*B*, *HCl*: prisms  $+ 8H_2O$  from  $H_2O$ . M.p.  $65-81^\circ$ . *B*, *HI*: prisms  $+ 6H_2O$  from  $H_2O$ . M.p.  $70-87^\circ$ .*Picrate*: yellow needles from  $H_2O$ . M.p.  $219^\circ$ . Spar. sol.  $H_2O$ ,  $EtOH$ .*Methiodide*: prisms  $+ 2H_2O$  from  $MeOH$ . M.p. about  $280^\circ$ . Spar. sol.  $H_2O$ .*N*-Benzoyl: prisms from  $EtOH$ . M.p.  $234-5^\circ$ . Sol.  $CHCl_3$ . Spar. sol.  $EtOH$ ,  $Me_2CO$ . Very spar. sol.  $H_2O$ ,  $Et_2O$ .*N*-Nitroso: prisms from  $EtOH$ . M.p.  $180-2^\circ$ . Sol. warm  $EtOH$ . Mod. sol.  $CHCl_3$ ,  $Me_2CO$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ . Insol. pet. ether.*Picrate*: yellow needles. Decomp. at  $285^\circ$ .Pyman, *J. Chem. Soc.*, 1909, 95, 1620; 1915, 107, 177.Pope, Gibson, *J. Chem. Soc.*, 1910, 97, 2209.Pyman, Reynolds, *ibid.*, 1324.**Pectolinarigenin** (5 : 7-Dihydroxy-6 : 4'-di-methoxyflavone) $C_{17}H_{14}O_6$ 

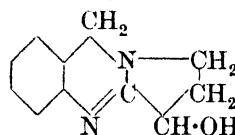
MW, 314

Yellow needles from  $EtOH$  or dil.  $Me_2CO$ . M.p.  $215-16^\circ$ .*Diacetyl*: needles. M.p.  $151^\circ$ .*7*-Me ether:  $C_{18}H_{16}O_6$ . MW, 328. Yellow needles. M.p.  $188^\circ$ . *Acetyl*: m.p.  $188^\circ$ .*Di*-Me ether:  $C_{19}H_{18}O_6$ . MW, 342. M.p.  $162^\circ$ .*Glucoside*: see Pectolinarin.Merz, Wu, *Arch. Pharm.*, 1936, 274, 126.  
Robinson, Schwarzenbach, *J. Chem. Soc.*, 1930, 829.**Pectolinarin** $C_{29}H_{34}O_{15}$ 

MW, 622

A glucoside from the flowers of *Linaria vulgaris*, Linn. M.p.  $240-50^\circ$  decomp. Hyd. by conc.  $HCl$  to glucose, rhamnose and pectolinarigenin.*Hepta-acetyl*: cryst. from  $Et_2O$ -pet. ether. M.p.  $134-8^\circ$ .  $[\alpha]_D^{18} - 68.5^\circ$ .

See first reference above.

**Peganine** (*Vasicine*) $C_{11}H_{12}ON_2$ 

MW, 188

*l*-.  
Found in *Adhatoda Vasica*, N. Cryst. from  $EtOH$ . M.p.  $211-12^\circ$ .  $[\alpha]_D^{18} - 254^\circ$  in  $CHCl_3$ ,  $- 61.5^\circ$  in  $EtOH$ .*dl*-.  
Needles from  $EtOH$ . M.p.  $209-10^\circ$  ( $196^\circ$ ). Sol.  $EtOH$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ ,  $Et_2O$ ,  $C_6H_6$ .*B*, *HCl*: needles  $+ 2H_2O$ . M.p. anhyd.  $208^\circ$ .*B*, *HI*: needles  $+ 2H_2O$ . M.p. anhyd.  $195^\circ$ .*Methiodide*: needles from  $MeOH$ . M.p.  $187^\circ$ .*Acetyl deriv.*: m.p.  $122-3^\circ$ . B.p.  $230-40^\circ/0.01$  mm.



*Picrate*: needles. M.p. 199° decomp.

Späth, Kuffner, Platzer, *Ber.*, 1935, **68**, 497, 699, 1384.

Späth, Platzer, *Ber.*, 1936, **69**, 255, 384.

Narang, Ray, *J. Chem. Soc.*, 1936, 686.

### Peimine

$C_{26}H_{43}O_3N$  MW, 417

Alkaloid from *Fritillaria Roylei* (Pei-Mu). M.p. 224°.

$B.HCl$ : m.p. 295° decomp.

$B.HBr$ : m.p. 293.5–294°.

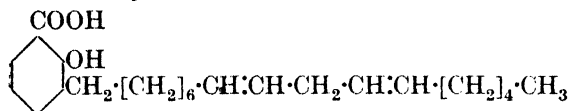
$B.H_2SO_4$ : m.p. 278–80°.

$B_2.H_2PtCl_6$ : m.p. 233–5° decomp.

$B.HAuCl_4$ : m.p. 164–5°.

Chi, Kao, Chang, *J. Am. Chem. Soc.*, 1936, **58**, 1306.

### Pelandjaic Acid (*Pentaspadonic acid*)



Suggested formula

$C_{24}H_{36}O_3$  MW, 372

Obtained from Minjak Pelandjau, an oily exudation from wood of *Pentaspadon molleyi*, H. Cryst. M.p. 25–6°.  $KMnO_4 \rightarrow$  azelaic and suberic acids.

*Me ester*:  $C_{25}H_{38}O_3$ . MW, 386. B.p. 225–35°/0.4 mm.  $D_4^{25}$  0.9170.  $n_D^{25}$  1.51477.

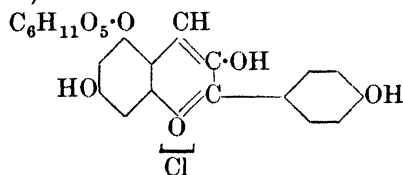
v. Romberg, v. Veen, *Chem. Abstracts*, 1930, **24**, 124.

v. Romberg, v. Veen, Smit, *Chem. Abstracts*, 1931, **25**, 937.

### Pelargone.

See Di-*n*-octyl Ketone.

**Pelargonenin chloride** (*Pelargonidin 5-β-glucoside*)



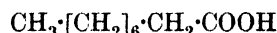
$C_{21}H_{21}O_{10}Cl$  MW, 468.5

Scarlet-red needles. Sol. MeOH. Mod. sol. EtOH. Spar. sol.  $H_2O$ , HCl. Acid sols.  $\rightarrow$  red col. with bluish fluor.  $Na_2CO_3$ . Aq.  $\rightarrow$  violet-blue col.

Willstätter, Bolton, *Ann.*, 1916, **412**, 136.

Léon, Robertson, Robinson, Seshardi, *J. Chem. Soc.*, 1931, 2673.

**Pelargonic Acid** (*Nonanoic acid, nonylic acid, nonic acid, octane-1-carboxylic acid*)



$C_9H_{18}O_2$  MW, 158

Oil. F.p. 12.5°. B.p. 253–4°/760 mm., 186°/100 mm.  $D_4^{12.5}$  0.9109,  $D_D^{17.5}$  0.9068.  $n_D$  1.43057. Spar. volatile in steam.

*Me ester*:  $C_{10}H_{20}O_2$ . MW, 172. B.p. 213–14°/756 mm.  $D^{17.5}$  0.8765.

*Et ester*:  $C_{11}H_{22}O_2$ . MW, 186. F.p. –36.7°. B.p. 216–19° (227°).  $D_4^0$  0.88156,  $D_4^{15}$  0.86920.

*2-Iodoethyl ester*: b.p. 169°/15 mm.

*2-Diethylaminoethyl ester*: m.p. 131°.

*tert.-Butyl ester*:  $C_{13}H_{26}O_2$ . MW, 214. Liq. B.p. 242°.

*Heptyl ester*:  $C_{16}H_{32}O_2$ . MW, 256. F.p. –15.54°. B.p. 210°/75 mm., 192.5°/30 mm.  $D_4^0$  0.8745,  $D_4^{20}$  0.8553.  $n_D^{20}$  1.4350.

*p-Chlorophenacyl ester*: m.p. 59°.

*p-Bromophenacyl ester*: m.p. 68.5°.

*p-Iodophenacyl ester*: m.p. 77°.

*Chloride*:  $C_9H_{17}OCl$ . MW, 176.5. F.p. –60.5°. B.p. 215.35°/760 mm., 98°/15 mm.  $D_4^0$  0.95901,  $D_4^{15}$  0.94633.

*Nitrile*:  $C_9H_{17}N$ . MW, 139. F.p. –34.2°. B.p. 224° (214–16°).  $D_4^0$  0.83314,  $D_4^{15}$  0.82207. Sol. EtOH, Et<sub>2</sub>O. Insol.  $H_2O$ .

*Amide*:  $C_9H_{19}ON$ . MW, 157. Cryst. M.p. 98.9° (92–3°). Insol. cold  $H_2O$ .

*Diethylamide*: b.p. 167–9°/16 mm.

*Isobutylamide*: m.p. 37–8°. B.p. 162°/6 mm.

*Anilide*: cryst. from EtOH. M.p. 57.5°.

*o-Toluidide*: m.p. 73°.

*p-Toluidide*: cryst. from EtOH. M.p. 84°.

*1-Naphthylamide*: cryst. from EtOH. M.p. 91°.

*Anhydride*: f.p. 16° (5°). B.p. 207°/15 mm.

Eichler, *Ber.*, 1879, **12**, 1888.

Krafft, *Ber.*, 1882, **15**, 1691.

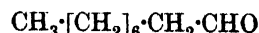
v. Braun, Sobiecki, *Ber.*, 1911, **44**, 1469.

Rochussen, *Chem. Abstracts*, 1930, **24**, 1755.

Deffet, *Chem. Abstracts*, 1932, **26**, 352.

Moses, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 2101.

**Pelargonic Aldehyde** (*Nonanal, nonanoic aldehyde, nonaldehyde, nonylic aldehyde*)



$C_9H_{18}O$  MW, 142

Found in mandarin and lemon-grass oil. B.p. 190–2°, 80–2°/13 mm.  $D^{15}$  0.8277.  $n_D^{15}$  1.42452. Polymerises readily with  $H_2SO_4$ . Oxidises slowly in air.

*Di-Me acetal*: b.p. 96–8°/15 mm.  $D_4^{18}$  0.8733.  $n_D^{18}$  1.4246.

*Di-Et acetal*: b.p. 130°/20 mm.

*Oxime*: leaflets from EtOH.Aq. M.p. 64°. Sol. most org. solvents. Volatile in steam.

*Semicarbazone*: cryst. from  $C_6H_6$ -pet. ether. or MeOH. M.p. 100°.

*Thiosemicarbazone*: m.p. 77°.

*Phenylsemicarbazone*: m.p. 131–2°.

*o-Tolylsemicarbazone*: m.p. 120–1°.

*p-Tolylsemicarbazone*: m.p. 155–6°.

*m-Nitrobenzoylhydrazone*: m.p. 87°.

*p-Nitrobenzoylhydrazone*: m.p. 103–4°.

*2:4-Dinitrophenylhydrazone*: m.p. 100°.

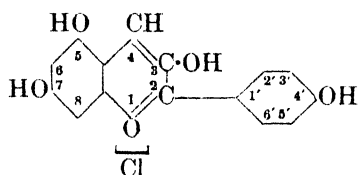
Strain, *J. Am. Chem. Soc.*, 1935, **57**, 758.

Wagner, *Chem. Abstracts*, 1928, **22**, 3131.

Bagard, *Bull. soc. chim.*, 1907, **1**, 351.

Sabatier, Mailhe, *Compt. rend.*, 1912, **154**, 563; 1906, **158**, 986.

### Pelargonidin chloride



$C_{15}H_{11}O_5Cl$

MW, 306.5

Leaflets from MeOH-Et<sub>2</sub>O, needles from MeOH-HCl. Does not melt below 350°. Sol. MeOH, EtOH. Mod. sol. H<sub>2</sub>O, CHCl<sub>3</sub>.

*5-Me ether*:  $C_{16}H_{13}O_5Cl$ . MW, 320.5. Cryst. Gives orange-red sol. in 0.5% HCl.

*7-Me ether*:  $C_{16}H_{13}O_5Cl$ . MW, 320.5. Solid. Gives orange-red sol. in 0.5% alc. HCl.

*3:4'-Di-Me ether*:  $C_{17}H_{15}O_5Cl$ . MW, 334.5. Crimson cryst. + H<sub>2</sub>O. M.p. 221° decomp.

*3-β-Glucoside*: Callistephin chloride, *q.v.*

*4'-β-Glucoside*: red plates. Decomp. at 184°. Sol. MeOH, EtOH. Spar. sol. cold H<sub>2</sub>O, Me<sub>2</sub>CO. *Picrate*: red prisms. Decomp. at 146–8°.

*5-β-Glucoside*: see Pelargoninenin chloride.

*7-β-Glucoside*: scarlet needles. Sol. H<sub>2</sub>O, EtOH, 0.5% HCl with deep red col.  $Na_2CO_3$ .Aq. → deep pink col. Alc. NaOH → violet-red col. *Picrate*: red plates. M.p. 180° decomp.

*3:5-Diglucoside*: see Pelargonin chloride.

*5-Benzoyl*: indigo plates. Sol. EtOH with violet col.  $Na_2CO_3$ .Aq. → violet col. *4'-Tetra-acetylglucoside*: reddish-brown needles.

Decomp. at 198°. AcOH → yellowish-red sol. with intense green fluor.  $Na_2CO_3$ .Aq. → bluish-pink sol. → blue with Me<sub>2</sub>CO.

*Picrate*: reddish plates with golden reflex.

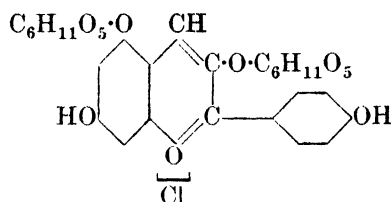
Willstätter, Burdick, *Ann.*, 1916, **412**, 163.

Robertson, Robinson, Sugiura, *J. Chem. Soc.*, 1928, 1533.

Léon, Robertson, Robinson, Seshadri, *J. Chem. Soc.*, 1931, 2673.

Nair, Robinson, *J. Chem. Soc.*, 1934, 1611.

### Pelargonin chloride (*Pelargonidin 3:5-diglucoside*)



$C_{27}H_{31}O_{15}Cl$

MW, 630.5

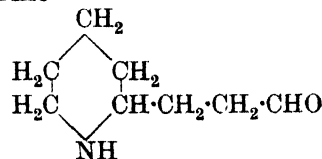
Fine red needles + 4H<sub>2</sub>O. M.p. anhyd. 180° decomp.  $[\alpha]_D - 244^\circ$ ,  $[\alpha]_{614} - 133^\circ$  in 0.1% HCl. Sol. H<sub>2</sub>O with orange-red sol. turning violet. Spar. sol. MeOH → red col. with green fluor. 20% HCl → pelargonidin + glucose.

Karrer, Widmer, *Helv. Chim. Acta*, 1927, **10**, 729.

Robinson, Todd, *J. Chem. Soc.*, 1932, 2488.

*Note*.—Robinson, Todd, (*loc. cit.*) have shown the identity of pelargonin, monardin (*q.v.*) and salvinin. Karrer and Meuron (*Helv. Chim. Acta*, 1932, **15**, 1214) have confirmed this. It is also possible that punicin may be identical with pelargonin.

### Pelletierine



$C_8H_{15}ON$

MW, 141

*dl.*

Occurs in root-bark of pomegranate tree (*Runica granatum*). Colourless oil. B.p.

106°/21 mm. Readily absorbs O from air, becoming dark coloured and resinous. Sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Alkaline in reaction.

$B, \text{HCl}$ : needles from  $\text{Me}_2\text{CO}$ . M.p. 143–4°.

$B, \text{HBr}$ : needles from  $\text{Me}_2\text{CO}$ . M.p. 140°.

*Picrate*: cryst. from  $\text{EtOH}$ . M.p. 150–1°.

*N-Acetyl*: b.p. 173–4°/18 mm.  $B, \text{HAuCl}_4$ : m.p. 95–6°.

*Oxime*: two forms. (i) From pet. ether. M.p. 96–7°; (ii) from  $\text{Et}_2\text{O}$ . M.p. 80°.

*Semicarbazone hydrochloride*: prisms from 50%  $\text{AcOH}$ . M.p. 188° decomp.

*Hydrazone*: b.p. 150°/20 mm.

*N-Carboethoxyl*: yellow oil. B.p. 173–4°/20 mm.

*N-Me*:  $\text{C}_9\text{H}_{17}\text{ON}$ . MW, 155. B.p. 98–102°/14 mm. Sol.  $\text{H}_2\text{O}$ .  $B, \text{HBr}$ : needles from  $\text{Me}_2\text{CO}$  or  $\text{C}_6\text{H}_6$ . M.p. 152°.

d.

*Sulphate*:  $[\alpha]_D^{25} + 5.86^\circ$  in  $\text{H}_2\text{O}$ .

d-Acid tartrate:  $[\alpha]_D^{20} + 21^\circ$  in  $\text{EtOH}$ .

l.

*Sulphate*:  $[\alpha]_D^{25} - 5.89^\circ$  in  $\text{H}_2\text{O}$ .

l-Acid tartrate:  $[\alpha]_D^{20} - 20.94^\circ$  in  $\text{EtOH}$ .

Hess, Eichel, *Ber.*, 1918, 51, 743.

Hess, *Ber.*, 1919, 52, 1005; 1917, 50, 373.

Note.—Tanret (*Compt. rend.*, 1920, 170, 1118) maintains that pelletierine occurs naturally in an optically active form,  $[\alpha]_D - 31.1^\circ$ .

*Sulphate*: m.p. 133°.  $[\alpha]_D - 30.3^\circ$ .

$B, \text{HCl}$ : m.p. 145°.  $[\alpha]_D - 41.2^\circ$ .

$B, \text{HBr}$ : m.p. 137°.  $[\alpha]_D - 32.5^\circ$ .

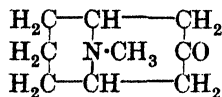
$B, \text{HNO}_3$ : m.p. 82–5°.  $[\alpha]_D - 34.8^\circ$ .

*N-Acetyl*: b.p. 205–10°/40 mm.  $[\alpha]_D + 32.6^\circ$ .

*Semicarbazone hydrochloride*: m.p. 168–70°.  $[\alpha]_D - 10.8^\circ$ .

This active pelletierine is stated to be sensitive to acids and alkalis, and rapidly racemises.

$\psi$ -Pelletierine (*N-Methylgranatonine*)



$\text{C}_9\text{H}_{15}\text{ON}$

MW, 153

Found in pomegranates. Prismatic plates from pet. ether. M.p. 48.5°. B.p. 246°/760 mm. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Less sol. pet.

ether. Sol. 2.5 parts  $\text{H}_2\text{O}$ . Strong base.  $\text{CrO}_3 \rightarrow$  intense green col.

$B, \text{HAuCl}_4$ : pale yellow cryst. from  $\text{H}_2\text{O}$ . M.p. 162° decomp. Sol. boiling  $\text{H}_2\text{O}$ .

*Oxime*: plates from  $\text{Et}_2\text{O}$ . M.p. 128–9°.

*Methiodide*: cryst. from  $\text{EtOH}$ . Aq. Does not melt below 280°.

*N-Oxide*: m.p. 160–2°.  $B, \text{HCl}$ : m.p. 224°.

*Picrate*: yellow needles from  $\text{H}_2\text{O}$ . M.p. 252–3° decomp.

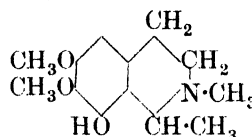
Ciamician, Silber, *Ber.*, 1892, 25, 1603; 1893, 26, 156, 2738.

Piccinini, Gazz. chim. ital., 1899, 29, i, 408; 1899, 29, ii, 312.

Hesse, *Ber.*, 1919, 52, 1011.

Menzies, Robinson, *J. Chem. Soc.*, 1924, 125, 2163.

**Pelletine** (*N-Methylanhalonidine*, 8-hydroxy-6:7-dimethoxy-1:2-dimethyl-1:2:3:4-tetrahydroisoquinoline)



$\text{C}_{13}\text{H}_{19}\text{O}_3\text{N}$

MW, 237

Found in *Anhalonium Williamsii*. Plates from  $\text{EtOH}$ . M.p. 111.5° (110°). Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Mod. sol. pet. ether. Spar. sol.  $\text{H}_2\text{O}$ . Aq. sol. reacts strongly alkaline. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow col.  $\rightarrow$  reddish-violet with  $\text{HNO}_3$ .  $\text{FeCl}_3 \rightarrow$  blue col.  $\rightarrow$  green on warming. Slightly narcotic. Used as hypnotic.

$B, \text{HI}$ : yellowish prisms. M.p. 125–30°. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ .

$B_2, \text{H}_2\text{PtCl}_6$ : m.p. 210–12°.

$B, \text{HAuCl}_4$ : orange cryst. M.p. 147–8° decomp.

*Methiodide*: plates or prisms +  $\text{H}_2\text{O}$  from  $\text{MeOH}$ . M.p. 199°. *Me ether*: prisms from  $\text{H}_2\text{O}$ . M.p. 225°. *Et ether*: m.p. 185–6°. *Benzyl ether*: m.p. 193–5°.

*Picrate*: yellow prisms from  $\text{H}_2\text{O}$ . M.p. 172 3°.

Hefter, *Ber.*, 1894, 27, 2975; 1901, 34, 3004.

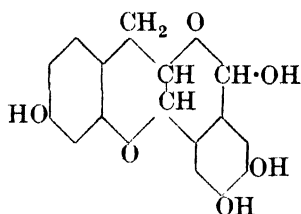
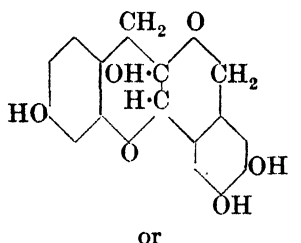
Späth, *Monatsh.*, 1921, 42, 97; *Ber.*, 1932, 65, 1778.

Späth, Boschan, *Monatsh.*, 1933, 63, 146.

Späth, Becke, *Ber.*, 1934, 67, 266.

Späth, Keszler, *Ber.*, 1936, 69, 755.

## Peltogynol

 $C_{15}H_{14}O_6$ 

MW, 302

Found in heart-wood of *Peltogyne porphyrocardia*. Prisms from  $H_2O$ . Becomes pink at  $200^\circ$ , dark red at  $240^\circ$  with decomp. and softening. Sol. MeOH, EtOH. Mod. sol.  $Me_2CO$ . Spar. sol. cold  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Aq. NaOH  $\rightarrow$  blue sol.  $\rightarrow$  bright red on standing. Alc.  $FeCl_3 \rightarrow$  blue col. Hot  $HNO_3 \rightarrow$  styphnic acid.

*Tri-Me ether*:  $C_{19}H_{20}O_6$ . MW, 344. Long needles or plates from AcOEt. M.p.  $198^\circ$ .  $[\alpha]_D^{25} + 250^\circ$  in  $CHCl_3$ .

*Tetra-Me ether*:  $C_{20}H_{22}O_6$ . MW, 358. Plates. M.p.  $175^\circ$ .  $[\alpha]_D^{20} + 264^\circ$  in  $CHCl_3$ .

*Tetra-acetyl*: needles from AcOH or  $C_6H_6$ -EtOH. M.p.  $173^\circ$ . Sol. AcOEt,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ , MeOH, EtOH,  $Et_2O$ , pet. ether.  $[\alpha]_D^{20} + 125^\circ$  in  $CHCl_3$ .

*Tetrabenzoyl*: plates from AcOH. M.p.  $244^\circ$ . Very sol.  $CHCl_3$ . Mod. sol. AcOEt.

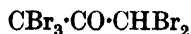
*Tetra-anisoyl*: plates from AcOH. M.p.  $218^\circ$  decomp. Sol.  $C_6H_6$ . Mod. sol.  $Me_2CO$ . Spar. sol. MeOH,  $Et_2O$ .

Robinson, Robinson, *J. Chem. Soc.*, 1935, 744.

## Penta-acetylglucose.

See under Glucose.

## Pentabromoacetone

 $C_3HOBr_5$ 

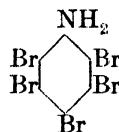
MW, 453

Needles from  $H_2O$  or EtOH, prisms from  $Et_2O$ . M.p.  $79-80^\circ$  ( $72^\circ$ ). Very sol. org.

solvents. Insol. cold  $H_2O$ . Sublimes. Volatile in steam.

Jackson, Adams, *J. Am. Chem. Soc.*, 1915, 37, 2533.

## Pentabromoaniline

 $C_6H_2NBr_5$ 

MW, 488

Needles from EtOH-toluene(1:2). M.p.  $261-2^\circ$ .

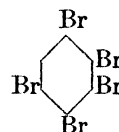
Jacobson, Löb, *Ber.*, 1900, 33, 705.

Hantzsch, Smythe, *ibid.*, 520.

## Pentabromoanisole.

See under Pentabromophenol.

## Pentabromobenzene

 $C_6HBr_5$ 

MW, 473

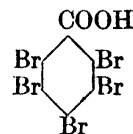
Needles from AcOH or EtOH. M.p.  $159-60^\circ$ . Mod. sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ , AcOH, ligroin. Very spar. volatile in steam.

Jacobson, Löb, *Ber.*, 1900, 33, 702.

Eckert, Steiner, *Monatsh.*, 1915, 36, 278.

Eckert, *J. prakt. Chem.*, 1921, 102, 362.

I.G., D.R.P., 595,461, (*Chem. Zentr.*, 1934, II, 1030).

Pentabromobenzoic Acid (*Perbromobenzoic acid*) $C_7HO_2Br_5$ 

MW, 517

Leaflets or needles from EtOH.Aq. M.p.  $252^\circ$  ( $234-5^\circ$ ). Sol. hot  $H_2O$ , EtOH. Spar. sol. pet. ether.

*Nitrile*:  $C_7NBr_5$ . MW, 498. Needles from  $C_6H_6$ -EtOH. Does not melt below  $300^\circ$ . Spar. sol. EtOH,  $Et_2O$ . Sublimes with slight decomp.

Merz, Weith, *Ber.*, 1883, 16, 2892.

Blanksma, *Chem. Zentr.*, 1912, II, 1965.

## Pentabromoethane

$\text{C}_2\text{HBr}_5$   $\text{CBr}_3\cdot\text{CHBr}_2$  MW, 425

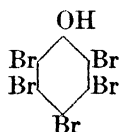
Prisms. M.p.  $56-7^\circ$  ( $54^\circ$ ). B.p.  $210^\circ/300$  mm. decomp. Very sol.  $\text{Et}_2\text{O}$ . Sol.  $\text{EtOH}$ .

Bourgoin, *Bull. soc. chim.*, 1875, **23**, 173.  
Hunter, Edgar, *J. Am. Chem. Soc.*, 1932, **54**, 2025.

## Pentabromophenetole.

See under Pentabromophenol.

## Pentabromophenol



$\text{C}_6\text{HOBr}_5$  MW, 489

Needles from  $\text{EtOH}$  or  $\text{CS}_2$ . M.p.  $229.5^\circ$  ( $225^\circ$ ). Sublimes.

*Me ether*: pentabromoanisole.  $\text{C}_7\text{H}_3\text{OBr}_5$ . MW, 503. Needles from  $\text{EtOH}$ . M.p.  $173-4^\circ$ .

*Et ether*: pentabromophenetole.  $\text{C}_8\text{H}_5\text{OBr}_5$ . MW, 517. Needles from  $\text{EtOH}$ . M.p.  $136^\circ$ .

*Propyl ether*:  $\text{C}_9\text{H}_7\text{OBr}_5$ . MW, 531. Needles from  $\text{EtOH}$ . M.p.  $98^\circ$ .

*Isopropyl ether*: needles from ligroin. M.p.  $86^\circ$ .

*n-Butyl ether*:  $\text{C}_{10}\text{H}_9\text{OBr}_5$ . MW, 545. M.p.  $79-80^\circ$ .

*sec.-n-Butyl ether*: m.p.  $57-8^\circ$ .

*Isobutyl ether*: needles from ligroin. M.p.  $92-3^\circ$ .

*Isoamyl ether*:  $\text{C}_{11}\text{H}_{11}\text{OBr}_5$ . MW, 559. Needles from ligroin. M.p.  $64-5^\circ$ .

*Allyl ether*:  $\text{C}_9\text{H}_5\text{OBr}_5$ . MW, 529. Needles from ligroin. M.p.  $167-8^\circ$ .

*Acetyl*: prisms from  $\text{EtOH}$ . M.p.  $197^\circ$  ( $171^\circ$ ).

Zincke, Birschel, *Ann.*, 1908, **362**, 227.

Bonneaud, *Bull. soc. chim.*, 1910, **7**, 776.

Kohn, Fink, *Monatsh.*, 1923, **44**, 187.

Lucas, Kemp, *J. Am. Chem. Soc.*, 1921, **43**, 1654.

Raiford, Howland, *J. Am. Chem. Soc.*, 1931, **53**, 1054.

## 1 : 1 : 1 : 2 : 2-Pentabromopropane

$\text{C}_3\text{H}_3\text{Br}_5$   $\text{CH}_3\cdot\text{CBr}_2\cdot\text{CBr}_3$  MW, 439

Needles from  $\text{C}_6\text{H}_6$ . M.p.  $212^\circ$ .

Loevenich, Loser, Dierichs, *Ber.*, 1927, **60**, 955.

## 1 : 1 : 2 : 3 : 3-Pentabromopropane

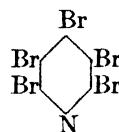
$\text{CHBr}_2\cdot\text{CHBr}\cdot\text{CHBr}_2$  MW, 439

Liq. B.p.  $165-75^\circ/17$  mm. ( $163-5^\circ/18$  mm.).

Prins, D.R.P., 261,689, (*Chem. Abstracts*, 1913, II, 394).

Mouneyrat, *Bull. soc. chim.*, 1898, **19**, 809.

## Pentabromopyridine

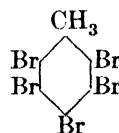


$\text{C}_5\text{NBr}_5$  MW, 474

Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $209.5-210^\circ$ .

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 940.

## Pentabromotoluene



$\text{C}_7\text{H}_3\text{Br}_5$  MW, 487

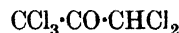
Needles from  $\text{C}_6\text{H}_6$ . M.p.  $282$  ( $280^\circ$ ). D<sup>17</sup> 2.97. Spar. sol.  $\text{EtOH}$ ,  $\text{AcOH}$ . Sol. about 102 parts  $\text{C}_6\text{H}_6$  at  $20^\circ$ .

Bodroux, Taboury, *Bull. soc. chim.*, 1912, **11**, 395.

Datta, Chatterjee, *J. Am. Chem. Soc.*, 1916, **38**, 2548.

Bodroux, *Ann. chim.*, 1929, **11**, 547.

## Pentachloroacetone



$\text{C}_3\text{HOCl}_5$  MW, 230.5

Liq. with odour resembling that of chloral. B.p.  $192^\circ/753$  mm. D<sup>18</sup> 1.69. Sol. 10 parts  $\text{H}_2\text{O}$ . Volatile in steam.

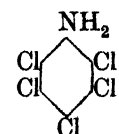
$\text{B}_4\text{H}_2\text{O}$ : plates. M.p.  $15-17^\circ$ .

Fritsch, *Ber.*, 1893, **26**, 598; *Ann.*, 1894, **279**, 317.

Staedler, *Ann.*, 1859, **111**, 293.

Buc, U.S.P., 1,391,758, (*Chem. Zentr.*, 1922, IV, 940).

## Pentachloroaniline



$\text{C}_6\text{H}_2\text{NCl}_5$  MW, 265.5

Needles from EtOH. M.p. 232°. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. ligroin.

N-Acetyl: N-Me, m.p. 136-7°. N-Et: m.p. 99-100°.

Willegerodt, Wilcke, *Ber.*, 1910, **43**, 2754.

Badische, D.R.P., 176,474, (*Chem. Zentr.*, 1907, I, 142).

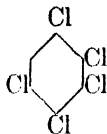
Durand, Huguenin, E.P., 217,753, (*Chem. Zentr.*, 1925, I, 301).

Quist, Salo, *Chem. Zentr.*, 1934, II, 594.

### Pentachloroanisole.

See under Pentachlorophenol.

### Pentachlorobenzene



C<sub>6</sub>HCl<sub>5</sub> MW, 250.5

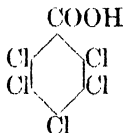
Needles from EtOH. M.p. 86° (84°). B.p. 275-7°. D<sup>10</sup> 1.8422, D<sup>16.5</sup> 1.8342. Mod. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, CCl<sub>4</sub>. Insol. cold EtOH.

Eckert, Steiner, *Ber.*, 1914, **47**, 2629.

v. der Linden, *Ber.*, 1912, **45**, 413.

Holleman, *Rec. trav. chim.*, 1920, **39**, 736.

### Pentachlorobenzoic Acid (Perchlorobenzoic acid)



C<sub>7</sub>HO<sub>2</sub>Cl<sub>5</sub> MW, 294.5

Needles. M.p. 201°. Sublimes in vacuo with slight decomp.

Me ester: C<sub>8</sub>H<sub>3</sub>O<sub>2</sub>Cl<sub>5</sub>. MW, 308.5. Cryst. from MeOH. M.p. 97°.

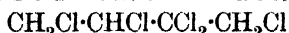
Chloride: C<sub>7</sub>OCl<sub>6</sub>. MW, 313. Cryst. from EtOH. M.p. 87°.

Steiner, *Monatsh.*, 1915, **36**, 827.

Merz, Weith, *Ber.*, 1883, **16**, 2885.

Kirpal, Kunze, *Ber.*, 1929, **62**, 2102.

### 1 : 2 : 2 : 3 : 4-Pentachlorobutane



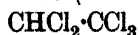
C<sub>4</sub>H<sub>5</sub>Cl<sub>5</sub> MW, 230.5

Liq. B.p. 85°/10 mm. D<sup>20</sup> 1.5543. n<sub>D</sub><sup>20</sup> 1.5157.

du Pont, U.S.P., 1,964,720, (*Chem. Zentr.*, 1934, II, 3180).

Carothers, Berehet, *J. Am. Chem. Soc.*, 1933, **55**, 1628.

### Pentachloroethane (Pentalin)



C<sub>2</sub>HCl<sub>5</sub> MW, 202.5

Liq. with odour of chloroform. F.p. -29° (-22°). B.p. 162.00°, 152.2°/644 mm., 93.6°/103 mm., 69.0°/37 mm. D<sup>15</sup> 1.6846, D<sup>25</sup> 1.6712. n<sub>D</sub><sup>15</sup> 1.50542. AlCl<sub>3</sub> → tetrachloroethylene.

Mouneyrat, *Bull. soc. chim.*, 1898, **19**, 260.

Salzbergwerk Neustassfurt, D.R.P., 248,982, (*Chem. Zentr.*, 1912, II, 299).

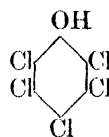
Timmermans, Martin, *J. chim. phys.*, 1926, **23**, 747.

Guyot, *Chimie et Industrie*, 1923, **10**, 13.

### Pentachlorophenetole.

See under Pentachlorophenol.

### Pentachlorophenol



C<sub>6</sub>HOCl<sub>5</sub> MW, 266.5

Cryst. + H<sub>2</sub>O from EtOH. needles from C<sub>6</sub>H<sub>6</sub>. M.p. 174°. anhyd. 191°. B.p. 309-10°/754.3 mm. D<sup>22</sup> 1.978. Very sol. EtOH, Et<sub>2</sub>O. Mod. sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold ligroin. Sublimes in long needles.

Me ether: pentachloroanisole. C<sub>7</sub>H<sub>3</sub>OCl<sub>5</sub>. MW, 280.5. Needles from MeOH. M.p. 108°. B.p. 289°/745 mm. decomp. Sol. EtOH. Sublimes readily.

Et ether: pentachlorophenetole. C<sub>8</sub>H<sub>5</sub>OCl<sub>5</sub>. MW, 294.5. Prisms from EtOH. M.p. 89-90°. Spar. sol. H<sub>2</sub>O.

Propyl ether: C<sub>9</sub>H<sub>7</sub>OCl<sub>5</sub>. MW, 308.5. Prisms from EtOH. M.p. 49-50°. Very sol. H<sub>2</sub>O.

Butyl ether: C<sub>10</sub>H<sub>9</sub>OCl<sub>5</sub>. MW, 322.5. Cryst. M.p. 15.5-16.5°. B.p. 343°.

Acetyl: needles from EtOH. M.p. 149.5-150.5° (147-8°). Sol. warm EtOH. Sublimes.

Propionyl: needles from ligroin. M.p. 78.5°. Very sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Butyryl: needles. M.p. 59-62°.

Benzoyl: prisms. M.p. 164-5° (159-60°).

Barral, *Bull. soc. chim.*, 1895, **13**, 342.

Weber, Wolff, *Ber.*, 1885, **18**, 335.

Biltz, Giese, *Ber.*, 1904, **37**, 4019.

Barral, Jambon, *Bull. soc. chim.*, 1900, **23**, 823.

Mathieson, McCombie, *J. Chem. Soc.*, 1931, 1110.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.

Tomcsik, *J. pharm. chim.*, 1930, **11**, 101.

I.G., D.R.P., 527,393, (*Chem. Zentr.*, 1931, II, 2785).

## 1 : 1 : 1 : 2 : 3-Pentachloropropane


 $\text{C}_3\text{H}_3\text{Cl}_5$  MW, 216.5

Needles from hot EtOH. M.p. 179–80°. Sublimes.

Vitoria, *Rec. trav. chim.*, 1905, **24**, 282.

Henry, *ibid.*, 342.

## 1 : 1 : 2 : 3 : 3-Pentachloropropane

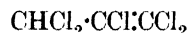

 $\text{C}_3\text{H}_3\text{Cl}_5$  MW, 216.5

Liq. B.p. 198–200°, 126°/90 mm.  $D_4^{25}$  1.6086.  $n_D^{18.5}$  1.5131.

Prins, *J. prakt. Chem.*, 1914, **89**, 421; D.R.P., 261,689, (*Chem. Zentr.*, 1913, II, 394).

Prins, Engelhard, *Rec. trav. chim.*, 1935, **54**, 307.

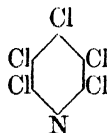
## 1 : 1 : 2 : 3 : 3-Pentachloropropylene


 $\text{C}_3\text{HCl}_5$  MW, 214.5

Liq. B.p. 183°, 116°/9 mm.  $D_4^{25}$  1.6317.  $n_D^{20}$  1.5313.

Prins, *J. prakt. Chem.*, 1914, **89**, 419; D.R.P., 261,689, (*Chem. Zentr.*, 1913, II, 394).

## Pentachloropyridine

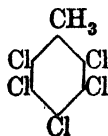

 $\text{C}_5\text{NCl}_5$  MW, 251.5

Plates or needles from EtOH. M.p. 124°. Mod. sol. hot EtOH. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Volatile in steam. Possesses no basic properties.

Sell, Dootson, *J. Chem. Soc.*, 1897, **71**, 1082; 1898, **73**, 441.

Hertog, Wibaut, Ley, *Rec. trav. chim.*, 1932, **51**, 381.

## Pentachlorotoluene


 $\text{C}_7\text{H}_3\text{Cl}_5$  MW, 264.5

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 218°. B.p. 301°. Sol. boiling C<sub>6</sub>H<sub>6</sub>. Spar. sol. CS<sub>2</sub>. Very spar. sol. EtOH, Et<sub>2</sub>O.

Beilstein, Kuhlberg, *Ann.*, 1869, **150**, 298. Fichter, Glantzstein, *Ber.*, 1916, **49**, 2486.

## Pentacosane


 $\text{C}_{25}\text{H}_{52}$  MW, 352

Pearly leaflets from EtOH. M.p. 55.5–56°.

Brigl, *Z. physiol. Chem.*, 1915, **95**, 179.

The following pentacosanes have also been described.—

1. From paraffin oil. M.p. 53.8–54°. B.p. 152.5°/0 mm.

Krafft, *Ber.*, 1907, **40**, 4783.

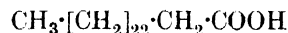
2. From paraffin oil. M.p. 54°. B.p. 282–4°/40 mm.  $D_4^{60}$  0.7911,  $D_4^{90}$  0.7854.

Mabery, *Am. Chem. J.*, 1905, **33**, 288.

3. From high-boiling tar. M.p. 53.5–54°. B.p. 254°/15 mm.  $D_4^{65}$  1.43202.  $n_D^{60}$  1.42624.

Gluud, *Ber.*, 1919, **52**, 1040, 1049.

## Pentacosanic Acid


 $\text{C}_{25}\text{H}_{50}\text{O}_2$  MW, 382

Cryst. from Me<sub>2</sub>CO. M.p. 84–5°.

Me ester: C<sub>26</sub>H<sub>52</sub>O<sub>2</sub>. MW, 396. Cryst. from Me<sub>2</sub>CO. M.p. 61–2°.

Et ester: C<sub>27</sub>H<sub>54</sub>O<sub>2</sub>. MW, 410. Cryst. from Me<sub>2</sub>CO. M.p. 58–9°. B.p. 216–17°/0.5 mm.

Nitrile: *n*-tetracosyl cyanide. C<sub>25</sub>H<sub>49</sub>N. MW, 363. Cryst. from Me<sub>2</sub>CO. M.p. 58–9°.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.

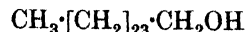
## Pentacosanol-1.

See Pentacosyl Alcohol.

## Pentacosanone-9.

See Octyl hexadecyl Ketone.

## Pentacosyl Alcohol (Pentacosanol-1)


 $\text{C}_{25}\text{H}_{52}\text{O}$  MW, 368

Cryst. from Me<sub>2</sub>CO. M.p. 78.5–79.5°. B.p. 214–16°/0.36 mm.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 916.

## Pentadecanal.

See Pentadecylic Aldehyde.

**Pentadecane** $\text{C}_{15}\text{H}_{32}$ 

MW, 212

Cryst. M.p.  $10^\circ$ . B.p.  $270.5^\circ/760$  mm.,  $194^\circ/100$  mm.,  $160^\circ/30$  mm.,  $144^\circ/15$  mm.  $D_4^{20}$  0.7689.

Krafft, *Ber.*, 1882, **15**, 1700.

Mai, *Ber.*, 1889, **22**, 2134.

**Pentadecane-1 : 1-dicarboxylic Acid.**

See Tetradecylmalonic Acid.

**Pentadecanoic Acid.**

See Pentadecylic Acid.

**Pentadecanoic Aldehyde.**

See Pentadecylic Aldehyde.

**Pentadecanol-1.**

See Pentadecyl Alcohol.

**Pentadecanol-3.**

See Ethyldodecylcarbinol.

**Pentadecanone-2.**

See Methyl tridecyl Ketone.

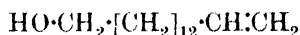
**Pentadecanone-3.**

See Ethyl dodecyl Ketone.

**Pentadecanone-8.**

See Caprylone.

**1-Pentadecenol-15** (*Pentadecylenic alcohol, 14-pentadecenyl alcohol, 15-hydroxy-1-pentadecylene*)

 $\text{C}_{15}\text{H}_{30}\text{O}$ 

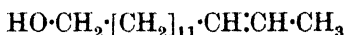
MW, 226

Leaflets from pet. ether. M.p.  $32-3^\circ$ . B.p.  $170-2^\circ/10$  mm.

*Phenylurethane* : leaflets from EtOH. M.p.  $60.5-61^\circ$ .

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, **10**, 128.

**2-Pentadecenol-15** (*13-Pentadecenol-1, iso-pentadecylenic alcohol, 13-pentadecenyl alcohol, 15-hydroxy-2-pentadecylene*)

 $\text{C}_{15}\text{H}_{30}\text{O}$ 

MW, 226

Leaflets from pet. ether. M.p.  $40-40.5^\circ$ . B.p.  $170-2^\circ/8$  mm.

*Phenylurethane* : leaflets from EtOH. M.p.  $68-68.4^\circ$ .

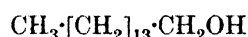
Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, **10**, 130.

**Pentadecenyl Alcohol.**

See Pentadecenol.

**Pentadecenylsalicylic Acid.**

See Ginkgolic Acid.

**Pentadecyl Alcohol** (*Pentadecanol-1*) $\text{C}_{15}\text{H}_{32}\text{O}$ 

MW, 228

Cryst. M.p.  $45-6^\circ$  ( $44^\circ$ ).

*Formyl* : cryst. M.p.  $13.69^\circ$ . B.p.  $201.5^\circ/30$  mm.  $D_4^{25}$  0.8618.  $n_D^{20}$  1.4399.

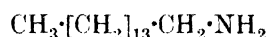
*Palmityl* : cryst. from EtOH. M.p.  $55.5^\circ$ .

*Phenylurethane* : plates from  $\text{C}_6\text{H}_6$ . M.p.  $72^\circ$ .

Simonini, *Monatsh.*, 1893, **14**, 85.

Gascard, *Ann. chim.*, 1921, **15**, 332.

Ruhoff, Reid, *J. Am. Chem. Soc.*, 1933, **55**, 3825.

**Pentadecylamine** (*1-Amino-n-pentadecane*) $\text{C}_{15}\text{H}_{33}\text{N}$ 

MW, 227

Leaflets or needles. M.p.  $36.5^\circ$  ( $33^\circ$ ). B.p.  $298-301^\circ$  ( $301^\circ/720$  mm.). Sol. EtOH,  $\text{Et}_2\text{O}$ .

*B,HCl* : plates from EtOH. M.p.  $199^\circ$ .

*Acetyl* : needles from pet. ether. M.p.  $72^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ , hot ligroin. Insol.  $\text{H}_2\text{O}$ .

Naegeli, Grüntuch, Lendorff, *Helv. Chim. Acta*, 1929, **12**, 240.

**3-Pentadecylcatechol.**

See Hydrourushiol.

**Pentadecylene-carboxylic Acid.**

See Gaidic Acid and Palmitoleic Acid.

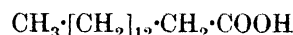
**Pentadecylenic Alcohol.**

See Pentadecenol.

**Pentadecyl *p*-hydroxyphenyl Ketone.**

See *p*-Hydroxypalmitophenone.

**Pentadecylic Acid** (*Pentadecanoic acid, tetradecane-1-carboxylic acid*)

 $\text{C}_{15}\text{H}_{30}\text{O}_2$ 

MW, 242

Plates from  $\text{Me}_2\text{CO.Aq.}$  M.p.  $53-4^\circ$  ( $51^\circ$ ). B.p.  $257^\circ/100$  mm. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether.

*Me ester* :  $\text{C}_{16}\text{H}_{32}\text{O}_2$ . MW, 256. Needles from EtOH.Aq. M.p.  $18.5^\circ$ .

*Et ester* :  $\text{C}_{17}\text{H}_{34}\text{O}_2$ . MW, 270. Needles from EtOH.Aq. M.p.  $14^\circ$ . Sol. most org. solvents.

*Chloride* :  $\text{C}_{15}\text{H}_{29}\text{OCl}$ . MW, 260.5. Liq. B.p.  $157^\circ/5$  mm.

*Amide* :  $\text{C}_{15}\text{H}_{31}\text{ON}$ . MW, 241. Needles from EtOH. M.p.  $102.5^\circ$ . Sol. boiling EtOH. Mod. sol.  $\text{Et}_2\text{O}$ . Insol. cold  $\text{H}_2\text{O}$ , pet. ether.

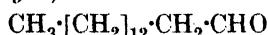
Le Sueur, *J. Chem. Soc.*, 1905, **87**, 1898.

Majima, Nakamura, *Ber.*, 1913, **46**, 4094.

Ford-Moore, Phillips, *Rec. trav. chim.*, 1934, **53**, 857.



**Pentadecylic Aldehyde** (*Pentadecanal, pentadecanoic aldehyde*)



$\text{C}_{15}\text{H}_{30}\text{O}$  MW, 226

Needles. M.p. 24–5°. B.p. 185°/25 mm., 172–6°/15 mm. Polymerises readily.

*Oxime*: needles from EtOH.Aq. M.p. 86°. Sol. Et<sub>2</sub>O. Spar. sol. EtOH, C<sub>6</sub>H<sub>6</sub>, pet. ether.

*Thiosemicarbazone*: cryst. from Et<sub>2</sub>O. M.p. 95–96.5°. Spar. sol. EtOH, Me<sub>2</sub>CO, AcOEt, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, pet. ether.

*p*-*Bromophenylhydrazone*: cryst. M.p. 40–50°. Unstable.

*p*-*Nitrophenylhydrazone*: yellow plates from EtOH. M.p. 94–5°. Sol. EtOH, Me<sub>2</sub>CO, AcOH, AcOEt, CCl<sub>4</sub>, C<sub>6</sub>H<sub>6</sub>.

*2:4-Dinitrophenylhydrazone*: m.p. 107.5°. Sol. AcOEt, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, hot Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH, CCl<sub>4</sub>.

*Benzoylhydrazone*: plates from EtOH. M.p. 87°.

*m*-*Nitrobenzoylhydrazone*: plates from EtOH. M.p. 102°. Spar. sol. Et<sub>2</sub>O.

*Cyanhydrin*: see under 1-Hydroxypalmitic Acid.

Le Sueur, *J. Chem. Soc.*, 1905, **87**, 1896.

Landa, *Bull. soc. chim.*, 1925, **37**, 1235.

### 3-Pentadecylphenol.

See Hydroginkgol.

### Pentadecyl phenyl Ketone.

See Palmitophenone.

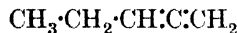
### 5-Pentadecylresorcinol.

See Hydrobilobol.

### 6-Pentadecylsalicylic Acid.

See Hydroginkgolic Acid.

**1:2-Pentadiene** (*Ethylallene, propylidene-ethylene*)

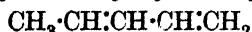


$\text{C}_5\text{H}_8$  MW, 68

Mobile liq. B.p. 44–5°.  $D_4^{20}$  0.6890.  $n_D^{20}$  1.4149.

Bouis, *Bull. soc. chim.*, 1927, **41**, 1160.

**1:3-Pentadiene** (*1-Methylbutadiene-1:3, 1-methyldivinyl, 1-methylerythrene, piperylene*)



$\text{C}_5\text{H}_8$  MW, 68

Liq. B.p. 42°.  $D_4^{15.6}$  0.6887,  $D_4^{20}$  0.6830.  $n_D^{15.6}$  1.43443,  $n_D^{20}$  1.4280.

Auwers, Westermann, *Ber.*, 1921, **54**, 2993.

Prévost, *Ann. chim.*, 1928, **10**, 173.

Farmer, Warren, *J. Chem. Soc.*, 1931, 3221.

**1:4-Pentadiene** (*Divinylmethane, allylethylene*)



$\text{C}_5\text{H}_8$  MW, 68

Liq. B.p. 25.8–26.2°/756 mm.  $D_4^{20}$  0.6594.  $n_D^{20}$  1.3883.

Kogerman, *J. Am. Chem. Soc.*, 1930, **52**, 5060.

Shoemaker, Boord, *J. Am. Chem. Soc.*, 1931, **53**, 1505.

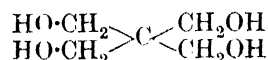
### 2:3-Pentadiene.

See sym.-Dimethylallene.

### 1:3-Pentadiene-1-carboxylic Acid.

See Sorbic Acid.

**Pentaerythritol** (*Tetrahydroxymethylmethane, tetramethylolmethane, tetrahydroxytetramethylmethane*)



$\text{C}_5\text{H}_{12}\text{O}_4$  MW, 136

Cryst. from dil. HCl. M.p. 260° (253°). Sol. 18 parts H<sub>2</sub>O at 15°. Heat of comb.  $\text{C}_p$  661.4 Cal.  $\text{HNO}_3 \longrightarrow$  oxalic acid.  $\text{CrO}_3 \longrightarrow \text{H} \cdot \text{COOH} + \text{CO}_2$ .

*Tetra-Et ether*:  $\text{C}_{13}\text{H}_{28}\text{O}_4$ . MW, 248. Oil. B.p. 220–5°.  $D_4^{20}$  0.9229,  $D_4^{16}$  0.9082.

*Tetra-acetyl*: needles from H<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>. M.p. 82–3°.

*Tetranitrate*: niperyt, penthit. Prisms from Me<sub>2</sub>CO–EtOH. M.p. 138–40°. Sol. Me<sub>2</sub>CO. Spar. sol. EtOH, Et<sub>2</sub>O. Reduces Fehling's.

Schwink, *Organic Syntheses*, Collective Vol. I, 417.

Stellbacher, *Chem. Zentr.*, 1919, II, 261.

Deutsche Gold- und Silber-Scheideanstalt v. Roessler, D.R.P., 596,509, (*Chem. Zentr.*, 1933, II, 2190).

Friederich, Brün, *Ber.*, 1930, **63**, 2681.

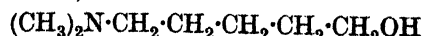
Bincer, Hess, *Ber.*, 1928, **61**, 539.

Clarke, U.S.P., 1,583,658, (*Chem. Zentr.*, 1926, II, 1191).

Desvergues, *Chem. Abstracts*, 1933, **27**, 4675.

Dumontel, Giovetti, *Industria chimica*, 1934, **9**, 767 (Review).

**Pentahomocholine** (*5-Dimethylamino-n-amyl alcohol*)



$\text{C}_7\text{H}_{17}\text{ON}$  MW, 131

Liq. B.p. 115–16°/25 mm. Misc. with H<sub>2</sub>O. Volatile in steam. Weak base.

*Methochloride*: cryst.

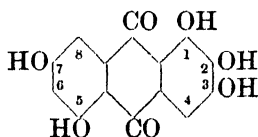
*Methiodide*: leaflets. M.p. 134°.

**1 : 2 : 3 : 5 : 7-Pentahydroxyanthraquinone**

*Methochloroplatinate* : pale yellow needles from EtOH.Aq. M.p. 213°. Very sol. H<sub>2</sub>O. Spar. sol. EtOH.

*Methochloroaurate* : brownish-yellow leaflets from H<sub>2</sub>O. M.p. 147°.

v. Braun, *Ber.*, 1916, **49**, 976.

**1 : 2 : 3 : 5 : 7-Pentahydroxyanthraquinone (5 : 7-Dihydroxyanthragallol)**

C<sub>14</sub>H<sub>8</sub>O<sub>7</sub>

MW, 288

Red cryst. from EtOH. Does not melt below 360°. Sol. boiling EtOH, Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O, AcOH. Insol. H<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin.

*Penta-acetyl* : pale yellow needles from EtOH. M.p. 229°. Sol. hot EtOH, AcOH.

Noah, *Ber.*, 1886, **19**, 751.

Liebermann, v. Kostanecki, Noah, *Ann.*, 1887, **240**, 273.

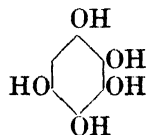
**1 : 2 : 4 : 5 : 8-Pentahydroxyanthraquinone (Alizarin Cyanine R)**

Bronze leaflets from PhNO<sub>2</sub>. Distils undecomposed. Conc. H<sub>2</sub>SO<sub>4</sub> → blue sol. with red fluor., + boric acid → greenish blue.

*Penta-acetyl* : leaflets from C<sub>6</sub>H<sub>6</sub>.

Bayer, D.R.P., 66,153.

Gattermann, *J. prakt. Chem.*, 1891, **43**, 250.

**Pentahydroxybenzene**

C<sub>6</sub>H<sub>6</sub>O<sub>5</sub>

MW, 158

Two compounds of this formula are described in the literature.

1. Cryst. from conc. HI. Very sol. H<sub>2</sub>O. Insol. org. solvents.

Wenzel, Weidel, *Chem. Zentr.*, 1903, **II**, 829.

2. Pale violet needles from AcOEt-C<sub>6</sub>H<sub>6</sub>. Blackens on heating. Sol. EtOH, Et<sub>2</sub>O, AcOEt. Spar. sol. H<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>. Alkalis → green sols. FeCl<sub>3</sub> → reddish-brown col.

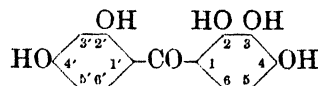
*Penta-acetyl* : needles from EtOH-pet. ether.

345

**2 : 4 : 6 : 3' : 5'-Pentahydroxybenzophenone**

M.p. 165° decomp. Sol. Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, EtOH, pet. ether.

Einhorn, Cobliner, Pfeiffer, *Ber.*, 1904, **37**, 122.

**2 : 3 : 4 : 2' : 4'-Pentahydroxybenzophenone**

C<sub>13</sub>H<sub>10</sub>O<sub>6</sub>

MW, 262

Yellowish needles + 2H<sub>2</sub>O from EtOH. M.p. 187° (168-70°). Sol. H<sub>2</sub>O → yellow col. → greenish-yellow → olive-green on standing.

Badische, D.R.Ps., 49,149, 50,451.

Atkinson, Heilbron, *J. Chem. Soc.*, 1926, 2690.

**2 : 3 : 4 : 3' : 4'-Pentahydroxybenzophenone**

Yellow needles + 2H<sub>2</sub>O from boiling H<sub>2</sub>O. M.p. anhyd. 192-3°. Sol. hot H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO, AcOH. Spar. sol. Et<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>. Conc. H<sub>2</sub>SO<sub>4</sub> → orange sol.

Noelting, Meyer, *Ber.*, 1897, **30**, 2591.

**2 : 3 : 4 : 3' : 5'-Pentahydroxybenzophenone**

*3 : 4 : 3' : 5'-Tetra-Me ether* : C<sub>17</sub>H<sub>18</sub>O<sub>6</sub>. MW, 318. Pale yellow cryst. from EtOH. M.p. 123-4°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. AcOH, ligroin. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 407.

**2 : 4 : 6 : 2' : 4'-Pentahydroxybenzophenone**

*Penta-Me ether* : C<sub>18</sub>H<sub>20</sub>O<sub>6</sub>. MW, 332. Pale yellow leaflets from EtOH.Aq. M.p. 138°.

Tambor, *Ber.*, 1910, **43**, 1888.

**2 : 4 : 6 : 2' : 6'-Pentahydroxybenzophenone**

*2' : 6'-Di-Me ether* : C<sub>15</sub>H<sub>14</sub>O<sub>6</sub>. MW, 290. Plates from EtOH.Aq. M.p. 216-18°. Sol. EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O. FeCl<sub>3</sub> → intense blue col.

Korczynski, Nowakowski, *Bull. soc. chim.*, 1928, **43**, 336.

**2 : 4 : 6 : 3' : 4'-Pentahydroxybenzophenone**

*See Maclurin.*

**2 : 4 : 6 : 3' : 5'-Pentahydroxybenzophenone**

*Penta-Me ether* : needles. M.p. 132-3°. Sol.

Et<sub>2</sub>O, AcOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, warm EtOH. Spar. sol. ligroin.

Mauthner, *J. prakt. Chem.*, 1913, 87, 409.

## 3 : 4 : 5 : 2' : 4'-Pentahydroxybenzophenone.

Yellow needles from EtOH.Aq. M.p. 242°. Sol. most org. solvents. Spar. sol. H<sub>2</sub>O. Alkalis → intense red col. Alc. FeCl<sub>3</sub> → green col.

3 : 4 : 5-Tri-Me ether : C<sub>16</sub>H<sub>16</sub>O<sub>6</sub>. MW, 304. Yellow needles from EtOH.Aq. or H<sub>2</sub>O. M.p. 165°. Conc. H<sub>2</sub>SO<sub>4</sub> → intense yellow sol.

Bargellini, Grippa, *Gazz. chim. ital.*, 1927, 57, 138.

Korczynski, Nowakowski, *Bull. soc. chim.*, 1928, 43, 335.

## 3 : 4 : 5 : 3' : 4'-Pentahydroxybenzophenone.

Yellow needles + 2H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 266°. Sol. hot H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO. Mod. sol. hot AcOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Conc. H<sub>2</sub>SO<sub>4</sub> → red col. → protocatechuic acid on heating.

Penta-Me ether : needles from EtOH. M.p. 119-20°. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. Oxime : needles from C<sub>6</sub>H<sub>6</sub>. M.p. 143°.

Noelting, Meyer, *Ber.*, 1897, 30, 2591.

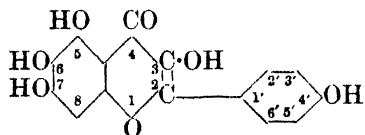
v. Kostanecki, Tambor, *Ber.*, 1906, 39, 4026.

Parkin, Weizmann, *J. Chem. Soc.*, 1906, 89, 1664.

## Pentahydroxycyclohexane.

See Quercitol.

## 3 : 5 : 6 : 7 : 4'-Pentahydroxyflavone



C<sub>15</sub>H<sub>10</sub>O<sub>7</sub>

MW, 302

Yellow micro-needles from AcOH.Aq. Darkens at 270°. Partially melts at 314-20° with decomp. Mg + acid alc. sol. → pinkish-red col. Alc. Pb(OAc)<sub>2</sub> → orange ppt. changing to greenish-brown. Alc. FeCl<sub>3</sub> → olive-green col.

3 : 6 : 7 : 4'-Tetra-Me ether : C<sub>19</sub>H<sub>18</sub>O<sub>7</sub>. MW, 358. Yellow plates from EtOH. M.p. 171°. Alc. FeCl<sub>3</sub> → olive-green col.

Penta-Me ether : see Tangeritin.

Goldsworthy, Robinson, *J. Chem. Soc.*, 1937, 48.

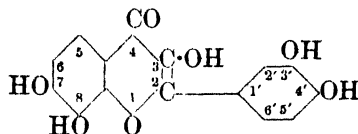
## 3 : 5 : 7 : 2' : 4'-Pentahydroxyflavone.

See Morin.

## 3 : 5 : 7 : 3' : 4'-Pentahydroxyflavone.

See Quercetin.

## 3 : 7 : 8 : 3' : 4'-Pentahydroxyflavone



C<sub>15</sub>H<sub>10</sub>O<sub>7</sub>

MW, 302

Pale yellow needles + H<sub>2</sub>O from EtOH.Aq. M.p. 308° decomp. Conc. H<sub>2</sub>SO<sub>4</sub> → intense yellow col. Dil. NaOH → reddish-yellow col.

7 : 8 : 3' : 4'-Tetra-Me ether : C<sub>19</sub>H<sub>18</sub>O<sub>7</sub>. MW, 358. Pale yellow needles from EtOH or AcOH-EtOH. M.p. 217°. Sol. hot AcOH. Spar. sol. EtOH. Conc. H<sub>2</sub>SO<sub>4</sub> → intense yellow col. Acetyl : needles from EtOH.Aq. M.p. 176°.

Penta-acetyl : needles from EtOH. M.p. 172-3°.

v. Kostanecki, Rudse, *Ber.*, 1905, 38, 938.

## 3 : 7 : 3' : 4' : 5'-Pentahydroxyflavone.

See Robinetin.

## 5 : 7 : 3' : 4' : 5'-Pentahydroxyflavone.

See Tricetin.

## 7 : 8 : 3' : 4' : 5'-Pentahydroxyflavone.

Yellow needles from EtOH.Aq. Does not melt below 345°.

Penta-acetyl : needles from EtOH. M.p. 263°.

Badhwar, Kang, Venkataraman, *J. Chem. Soc.*, 1932, 1110.

## Pentaketocyclopentane.

See Leuconic Acid.

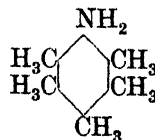
## Pental.

2-Methylbutylene-2, *q.v.*

## Pentalin.

See Pentachloroethane.

**Pentamethylaniline** (*Aminopentamethylbenzene*)



C<sub>11</sub>H<sub>17</sub>N

MW, 163

Prismatic cryst. from EtOH. M.p. 151-2°. B.p. 277-8°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

N-Me : C<sub>12</sub>H<sub>19</sub>N. MW, 177. Cryst. from EtOH. M.p. 60-1°.

N-Di-Me : C<sub>13</sub>H<sub>21</sub>N. MW, 191. Cryst. M.p. 53-4°.

N-Formyl : needles from EtOH.Aq. M.p. 217°.

**N-Acetyl**: needles from EtOH. M.p. 213.

Hoffmann, *Ber.*, 1885, **18**, 1822.

Limpach, *Ber.*, 1888, **21**, 645.

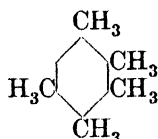
Willstätter, Kubli, *Ber.*, 1909, **42**, 4162.

Dimroth, Leichtlin, Friedemann, *Ber.*, 1917, **50**, 1543.

**Pentamethylanisole.**

See under Pentamethylphenol.

**Pentamethylbenzene**



$C_{11}H_{16}$

MW, 148

Prisms from EtOH.Aq. or EtOH- $C_6H_6$ . M.p. 53° (51.5°). B.p. 231°. Very sol. EtOH. Heat of comb.  $C_6$  1551.8 Cal.

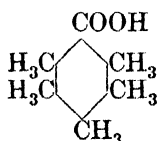
**Picrate**: golden-yellow prisms from EtOH. M.p. 131°.

Jacobsen, *Ber.*, 1887, **20**, 896.

v. Braun, Nelles, *Ber.*, 1934, **67**, 1099.

Smith, *Organic Syntheses*, 1930, **X**, 34.

**Pentamethylbenzoic Acid**



$C_{12}H_{16}O_2$

MW, 192

Needles from boiling  $H_2O$ ; leaflets or needles from EtOH.Aq. M.p. 210.5°. Very sol. hot EtOH. Insol. cold  $H_2O$ . Volatile in steam. Sublimes. Fuming  $HNO_3$  at 200°  $\rightarrow$  pentamethylbenzene +  $CO_2$ .

**Me ester**:  $C_{13}H_{18}O_2$ . MW, 206. Plates from MeOH. M.p. 67.5°. B.p. 299-300°.

**Amide**:  $C_{12}H_{17}ON$ . MW, 191. Leaflets from EtOH. M.p. 206°. Insol.  $H_2O$ .

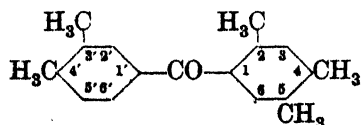
**Nitrile**:  $C_{12}H_{15}N$ . MW, 173. Needles from EtOH. M.p. 170° (168°). B.p. 294-5° (290-2°). Very sol. hot EtOH. Insol.  $H_2O$ .

Jacobsen, *Ber.*, 1889, **22**, 1221.

Hoffmann, *Ber.*, 1885, **18**, 1825.

Clement, *Compt. rend.*, 1934, **198**, 666.

**2 : 4 : 5 : 3' : 4'-Pentamethylbenzophenone**



$C_{18}H_{20}O$

MW, 252

Prisms from  $C_6H_6$ . M.p. 90°. B.p. 189-90°/3 mm. Very sol. most solvents.

Morgan, Coulson, *J. Chem. Soc.*, 1931, 2327.

**2 : 4 : 6 : 3' : 5'-Pentamethylbenzophenone.**

Prisms from pet. ether. M.p. 84-5°. Sol. hot EtOH. Conc.  $H_2SO_4 \rightarrow$  yellow sol.

Weiler, *Ber.*, 1899, **32**, 1910; 1900, **33**, 344.

**Pentamethylene.**

See Cyclopentane.

**Pentamethylene-aniline.**

See N-Phenylpiperidine.

**Pentamethylene bromide.**

See 1 : 5-Dibromo-n-pentane.

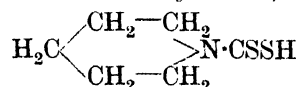
**Pentamethylene chloride.**

See 1 : 5-Dichloro-n-pentane.

**Pentamethylenediamine.**

See Cadaverine.

**Pentamethylenedithiocarbamic Acid**  
(Piperidine N-dithiocarboxylic acid)



$C_6H_{11}NS_2$

MW, 161

Free acid not known. Salts are powerful vulcanisation accelerators.

**Piperidine salt**: leaflets from EtOH. M.p. 174°. Well known rapid vulcanisation accelerator.

**Phenylhydrazine salt**: m.p. 218°.

**Me ester**:  $C_7H_{13}NS_2$ . MW, 175. Plates. M.p. 33-4°. B.p. about 260°. Very sol.

**Phenyl ester**:  $C_{12}H_{15}NS_2$ . MW, 237. M.p. 116-17°.

**p-Tolyl ester**:  $C_{13}H_{17}NS_2$ . MW, 251. M.p. 118-19°.

Losanitsch, *Ber.*, 1907, **40**, 2974.

Delépine, *Bull. soc. chim.*, 1902, **27**, 592.

Maas, Wolfenstein, *Ber.*, 1898, **31**, 2689.

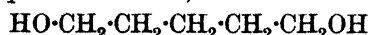
Clifford, Lichty, *J. Am. Chem. Soc.*, 1932, **54**, 1166.

I.C.I., E.P., 358,230, (*Brit. Chem. Abstracts*, 1932, 138).

**Pentamethyleneglycine.**

See Piperidinoacetic Acid.

**Pentamethylene Glycol** (1 : 5-Dihydroxypentane, pentandiol-1 : 5)



$C_5H_{12}O_2$

MW, 104

Oily liq. with bitter taste. B.p. 238-9° (260°) 155°/31 mm., 134°/12 mm., 119-20°/3

mm. Sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{Et}_2\text{O}$ .  $D^{20}_D$  1.004,  $D^{15}_D$  0.999,  $D^{20}_D$  0.9939.  $n^{20}_D$  1.4499.

*Di-Me ether*:  $\text{C}_7\text{H}_{16}\text{O}_2$ . MW, 132. B.p.  $156-7^\circ/760$  mm. ( $159^\circ$ ).  $D^{15}_D$  0.8616.  $n^{15}_D$  1.4094.

*Di-isoamyl ether*:  $\text{C}_{15}\text{H}_{32}\text{O}_2$ . MW, 244. Liq. B.p.  $276-7^\circ/759$  mm.,  $159-60^\circ/20$  mm.  $D^{18}_D$  0.844.

*Phenyl ether*:  $\text{C}_{11}\text{H}_{16}\text{O}_2$ . MW, 180. B.p.  $150-5^\circ/11$  mm. *Phenylurethane*: m.p.  $93^\circ$ .

*Diacetyl*: liq. with fruity odour. F.p.  $2^\circ$ . B.p.  $241^\circ/760$  mm.,  $122-3^\circ/3$  mm.  $D^{18}_D$  1.0328,  $D^{18}_D$  1.021.  $n^{15}_D$  1.4282,  $n^{19}_D$  1.4261.

*Succinyl*: m.p.  $19^\circ$ . B.p.  $88-9^\circ/1$  mm.  $D^{18}_D$  1.1373.  $n^{60}_D$  1.4583. *Dimeric form*: m.p.  $87^\circ$ .

*Di-p-nitrobenzoyl*: cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $104-5^\circ$ .

*Carbonate*: powder. M.p.  $44-6^\circ$ . Sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ , AcOH,  $\text{C}_6\text{H}_6$ . Insol. EtOH,  $\text{Et}_2\text{O}$ , pet. ether. *Dimeric form*: m.p.  $117-18^\circ$ .

*Di-phenylurethane*: needles from EtOH or EtOH- $\text{CHCl}_3$ . M.p.  $176^\circ$ .

*Di-1-naphthylurethane*: plates. M.p.  $147^\circ$ .

du Pont, U.S.P., 1,995,291, (*Chem. Abstracts*, 1935, 27, 2975).

Spanagel, Carothers, *J. Am. Chem. Soc.*, 1935, 57, 931.

Paul, *Bull. soc. chim.*, 1934, 1, 971.

Wojcik, Adams, *J. Am. Chem. Soc.*, 1933, 55, 4941.

Bennett, Heathcoat, *J. Chem. Soc.*, 1929, 273.

Müller, Rölz, Gero, *Monatsh.*, 1928, 50, 107.

v. Braun, Deutsch, Schmatloch, *Ber.*, 1912, 45, 1250.

Hamonet, *Bull. soc. chim.*, 1905, 33, 529.

v. Braun, Steindorff, *Ber.*, 1905, 38, 959.

Lespieau, *Ann. chim. phys.*, 1912, 27, 175.

### Pentamethyleneimine.

See Piperidine.

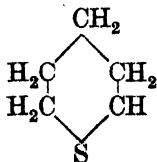
### Pentamethylene iodide.

See 1:5-Di-iodo-*n*-pentane.

### Pentamethylene oxide.

See Tetrahydropyran.

### Pentamethylene sulphide (Tetrahydrothiopyran)



$\text{C}_5\text{H}_{10}\text{S}$

MW, 102

Found in crude Mexican petroleum. Cryst. M.p.  $13^\circ$ . B.p.  $141^\circ/755$  mm. ( $141.5-142^\circ/747$  mm.). Sol. most org. solvents. Insol.  $\text{H}_2\text{O}$ .  $D^{15}_D$  0.9889,  $D^{18}_D$  0.9943,  $D^{20}_D$  0.9849.  $n^{18}_D$  1.5046. Stable at  $200^\circ$ . Volatile in steam. Tetra-nitromethane in EtOH  $\rightarrow$  golden-yellow col. Dil.  $\text{HNO}_3 \rightarrow$  pentamethylene sulphoxide.  $\text{KMnO}_4 \rightarrow$  pentamethylene sulphone.

$\text{B,HgCl}_2$ : leaflets from EtOH. M.p.  $137.5^\circ$ . Sol. hot EtOH. Insol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ .

*Methiodide*: needles from EtOH. Sublimes at  $192^\circ$  ( $162^\circ$ ) without melting. Sol.  $\text{H}_2\text{O}$ .

Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin.

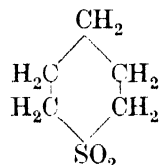
*Methochloroplatinate*: m.p.  $225^\circ$ . Sol.  $\text{H}_2\text{O}$ .

Clarke, *J. Chem. Soc.*, 1912, 101, 1805.

v. Braun, Trümpler, *Ber.*, 1910, 43, 548.

Grishkewitsch - Trochimowski, *Chem. Zentr.*, 1923, I, 1503.

### Pentamethylene sulphone



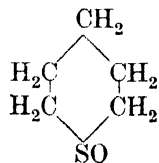
$\text{C}_5\text{H}_{10}\text{O}_2\text{S}$

MW, 134

Cryst. from  $\text{H}_2\text{O}$ . M.p.  $98.5-99^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH. Less sol.  $\text{Et}_2\text{O}$ . Insol. ligroin.

Grishkewitsch - Trochimowski, *Chem. Zentr.*, 1923, I, 1503.

### Pentamethylene sulphoxide



$\text{C}_5\text{H}_{10}\text{OS}$

MW, 118

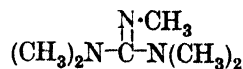
Yellow liq. which solidifies to glassy mass in exsiccator.

See previous reference.

### Pentamethylene-urethane.

See under Piperidine-*N*-carboxylic Acid.

### Pentamethylguanidine



$\text{C}_6\text{H}_{15}\text{N}_3$

MW, 129

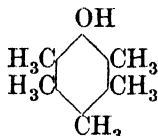
Liq. B.p.  $155-60^\circ$ . Sol.  $\text{H}_2\text{O}$ , org. solvents. Very hygroscopic. Fumes slightly in moist air. Readily absorbs  $\text{CO}_2$ . Strong base.

$\text{B,HgAuCl}_4$ : needles from  $\text{H}_2\text{O}$ . M.p.  $130-2^\circ$ .

*Picrate*: yellow needles from  $\text{H}_2\text{O}$ . M.p.  $165-6^\circ$  decomp.

Schenck, *Z. physiol. Chem.*, 1912, **77**, 386.  
 Lecher, *Graf, Ber.*, 1923, **56**, 1329.

**Pentamethylphenol** (*Hydroxypentamethylbenzene*)



$\text{C}_{11}\text{H}_{16}\text{O}$

MW, 164

Needles from EtOH or pet. ether. M.p.  $126^\circ$ . B.p.  $267^\circ$ . Gives no col. with  $\text{FeCl}_3$ .

*Me ether*: pentamethylanisole.  $\text{C}_{12}\text{H}_{18}\text{O}$ . MW, 178. Needles from EtOH. M.p.  $63-4^\circ$ . Sol. EtOH.

*Benzoyl*: rhombic plates from EtOH. M.p.  $127^\circ$ .

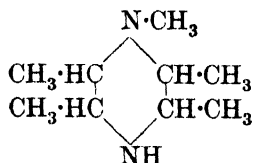
*Phenylurethane*: needles from EtOH-pet. ether. M.p.  $215^\circ$ .

Hey, *J. Chem. Soc.*, 1931, 1593.

Dimroth, Leichtlin, Friedemann, *Ber.*, 1917, **50**, 1543.

Hofmann, *Ber.*, 1885, **18**, 1826.

**2 : 3 : 4 : 5 : 6-Pentamethylpiperazine**  
 (2 : 3 : 4 : 5 : 6-Pentamethylhexahydropyrazine)



$\text{C}_9\text{H}_{20}\text{N}_2$

MW, 156

*$\gamma$ -Form*:

Prisms. M.p.  $45^\circ$ . B.p.  $201-2^\circ$ . Very hygroscopic.

*B, H<sub>2</sub>O*: prisms from  $\text{Me}_2\text{CO}$ . M.p.  $73-4^\circ$ . Sol. EtOH, AcOEt. Spar. sol.  $\text{C}_6\text{H}_6$ ,  $\text{Et}_2\text{O}$ .

*B, 2HCl*: needles. M.p. about  $300^\circ$ . Sol.  $\text{H}_2\text{O}$ . Insol. EtOH.

*B, HI*: prisms from  $\text{H}_2\text{O}$  or EtOH. M.p.  $161-2^\circ$ . Very sol.  $\text{H}_2\text{O}$ , hot EtOH.

*B, 2HI*: prisms from EtOH. M.p.  $240^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ , EtOH, MeOH. Almost insol.  $\text{Me}_2\text{CO}$ , AcOEt.

*N-p-Toluenesulphonyl*: prisms from EtOH, needles from pet. ether. M.p.  $124^\circ$ . *B, HCl*: prisms from EtOH. M.p.  $250^\circ$ . *d-Tartrate*: needles from  $\text{Me}_2\text{CO}$ . M.p.  $174-5^\circ$ .  $[\alpha]_{5461} + 9.7^\circ$  in  $\text{H}_2\text{O}$ . *d-Camphor-10-sulphonate*: prisms from  $\text{H}_2\text{O}$ . M.p.  $208-11^\circ$ .  $[\alpha]_{5461} + 28.4^\circ$  in  $\text{CHCl}_3$ .

*N-Nitroso*: pale yellow oil. M.p.  $24-5^\circ$ . B.p.  $155-7^\circ/15$  mm. Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ , pet. ether. Less sol. hot than cold  $\text{H}_2\text{O}$ . *d-Camphor-10-sulphonate*: cryst. from  $\text{Me}_2\text{CO}$ . M.p.  $213-15^\circ$  decomp.  $[\alpha]_{5461} + 15.2^\circ$  in  $\text{H}_2\text{O}$ .

*d-Tartrate*: cryst. from MeOH. M.p.  $164-5^\circ$ .  $[\alpha]_{5461} + 20.3^\circ$  in  $\text{H}_2\text{O}$ .

*Di-d-camphor-10-sulphonate*: cryst. from EtOH. M.p.  $261^\circ$ .  $[\alpha]_{5461} + 20.0^\circ$  in  $\text{H}_2\text{O}$ .

*Phenylthiourea*: prisms from pet. ether. M.p.  $154^\circ$ . Sol. EtOH.  $\text{C}_6\text{H}_6$ . Spar. sol. pet. ether.

*$\beta$ -Form*:

*p-Toluenesulphonyl*: prisms from EtOH or pet. ether. M.p.  $100-1^\circ$ .

*Phenylthiourea*: prisms from pet. ether. M.p.  $95^\circ$ .

Kipping, *J. Chem. Soc.*, 1932, 1340; 1933, 143.

**Pentanal.**

See Valeraldehyde.

**1-Pentanalone-2.**

See Propylglyoxal.

**1-Pentanalone-4.**

See Levulinic Aldehyde.

**Pentandiol.**

See Dihydroxypentane and Pentamethylene Glycol.

**Pentandione-2 : 3.**

See Acetylpropionyl.

**Pentandione-2 : 4.**

See Acetylacetone.

**n-Pentane**



$\text{C}_5\text{H}_{12}$

MW, 72

Found in American petroleum. F.p.  $-129^\circ$ . B.p.  $36.00^\circ$ .  $D_4^{20}$  0.62632.  $n_D^{20}$  1.35769. Sol. 200 parts  $\text{H}_2\text{O}$  at  $16^\circ$ .

Noller, *Organic Syntheses*, 1931, XI, 84.

Shepard, Henne, Midgley, *J. Am. Chem. Soc.*, 1931, **53**, 1948.

**Pentane-1-carboxylic Acid.**

See n-Caproic Acid.

**Pentane-2-carboxylic Acid.**

See 1-Methylvaleric Acid.

**Pentane-3-carboxylic Acid.**

See Diethylacetic Acid.

**Pentane-1 : 1-dicarboxylic Acid.**

See n-Butylmalonic Acid.

**Pentane-1 : 2-dicarboxylic Acid.**

See Propylsuccinic Acid.

**Pentane-1 : 4-dicarboxylic Acid.**

See 1-Methyladipic Acid.

**Pentane-1 : 5-dicarboxylic Acid.**

See Pimelic Acid.

**Pentane-2 : 2-dicarboxylic Acid.**

See Methylpropylmalonic Acid.

**Pentane-2 : 3-dicarboxylic Acid.**

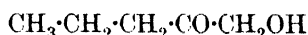
See 1-Methyl-2-ethylsuccinic Acid.

**Pentane-2 : 4-dicarboxylic Acid.**

See 1 : 3-Dimethylglutaric Acid.

**Pentane-3 : 3-dicarboxylic Acid.**

See Diethylmalonic Acid.

**Pentanol.**See *n*-Amyl Alcohol, Methyl-*n*-propylcarbinol, and Diethylcarbinol.**1-Pentanolone-2** (Butyrylcarbinol, 2-keto-*n*-amyl alcohol) $\text{C}_5\text{H}_{10}\text{O}_2$  MW, 102Oil. B.p. 54–6°/11 mm.  $D_4^{20}$  0.9860.  $n_D^{20}$  1.4234.

*Me ether*:  $\text{C}_6\text{H}_{12}\text{O}_2$ . MW, 116. B.p. 146–8° (152–3°/745 mm.), 117°/175 mm.  $D_4^{20}$  0.9139.  $n_D^{20}$  1.4119. Semicarbazone: needles. M.p. 97–8°. Very sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ , AcOEt,  $\text{C}_6\text{H}_6$ .  
 2 : 4-Dinitrophenylhydrazone: m.p. 128.5–129°.

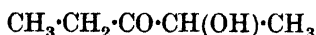
*Et ether*:  $\text{C}_7\text{H}_{14}\text{O}_2$ . MW, 130. B.p. 167°, 64–5°/17 mm., 60°/11 mm.  $D_4^{16}$  0.9218. Spar. sol.  $\text{H}_2\text{O}$ . Reduces  $\text{NH}_3 \cdot \text{AgNO}_3$ . Forms bisulphite comp. Semicarbazone: m.p. 86–7°.

2 : 4-Dinitrophenylhydrazone: m.p. 163°.

Osazone: m.p. 110–11°.

Henze, Rigler, *J. Am. Chem. Soc.*, 1934, **56**, 1350.Maruyama, *Chem. Abstracts*, 1933, **27**, 1863.Sommelet, *Bull. soc. chim.*, 1911, **9**, 36.Schmidt, Ascherl, *Ber.*, 1925, **58**, 358.**1-Pentanolone-4.**

See 3-Acetopropyl Alcohol.

**2-Pentanolone-3** (Methylpropionylcarbinol, 1-hydroxydiethyl ketone) $\text{C}_5\text{H}_{10}\text{O}_2$  MW, 102B.p. 152.5°/761 mm., 63°/20 mm., 45–8°/11 mm.  $D_4^{20}$  0.9742.  $n_D^{20}$  1.4218. Vigorously reduces  $\text{NH}_3 \cdot \text{AgNO}_3$  and Fehling's.*Me ether*:  $\text{C}_6\text{H}_{12}\text{O}_2$ . MW, 116. B.p. 133°/729 mm.*Et ether*:  $\text{C}_7\text{H}_{14}\text{O}_2$ . MW, 130. B.p. 145°/727 mm.*Butyl ether*:  $\text{C}_9\text{H}_{18}\text{O}_2$ . MW, 158. B.p. 80–2°/35 mm.  $D_4^{20}$  0.8693.  $n_D^{20}$  1.4143.

Semicarbazone: m.p. 208–9°.

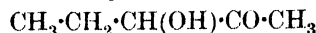
Phenylhydrazone: needles from EtOH.Aq.

M.p. 107°. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{C}_6\text{H}_6$ .

Osazone: m.p. 167°.

Vénus-Daniloff, *Bull. soc. chim.*, 1928, **43**, 582.Henze, Murchison, *J. Am. Chem. Soc.*, 1933, **55**, 4257.Schmidt, Ascherl, *Ber.*, 1925, **58**, 357.Gauthier, *Ann. chim. phys.*, 1909, **16**, 324; *Compt. rend.*, 1911, **152**, 1101.**2-Pentanolone-4.**

See Acetoisopropyl Alcohol.

**3-Pentanolone-2** (Ethylacetylcarbinol, 1-acetopropyl alcohol, methyl 1-hydroxypropyl ketone) $\text{C}_5\text{H}_{10}\text{O}_2$  MW, 102B.p. 147–8°/761 mm., 77°/35 mm., 59–59.5°/27 mm.  $D_4^{20}$  0.9500. Misc. with  $\text{H}_2\text{O}$ , EtOH, and most other org. solvents.

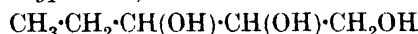
*Et ether*:  $\text{C}_7\text{H}_{14}\text{O}_2$ . MW, 130. B.p. 76°/67 mm.  $D_4^{17}$  0.8849.  $n_D^{14}$  1.4075. Semicarbazone: m.p. 93–5°.

Semicarbazone: m.p. 216–17°.

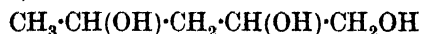
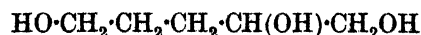
Osazone: m.p. 167°.

Grard, *Ann. chim.*, 1930, **13**, 336.Vénus-Daniloff, *Bull. soc. chim.*, 1928, **43**, 582.Bouis, *Bull. soc. chim.*, 1932, **51**, 1177.**Pentanone.**

See Diethyl Ketone and Methyl propyl Ketone.

**Pentantriol-1 : 2 : 3** (1-Ethyl glycerol, 1 : 2 : 3-trihydroxypentane) $\text{C}_5\text{H}_{12}\text{O}_3$  MW, 120Syrup. B.p. 192°/63.3 mm.  $D_4^{20}$  1.0851. Misc. with  $\text{H}_2\text{O}$ , EtOH. Sol.  $\text{Et}_2\text{O}$ . Sweet taste.

Tribenzoyl: m.p. 99–100°.

Wagner, *Ber.*, 1888, **21**, 3349.Delaby, *Compt. rend.*, 1923, **176**, 589.**Pentantriol-1 : 2 : 4** (1 : 2 : 4-Trihydroxypentane) $\text{C}_5\text{H}_{12}\text{O}_3$  MW, 120Liq. B.p. 180°/27 mm.  $D_4^{20}$  1.135,  $D_4^{22}$  1.120.Wagner, *Ber.*, 1888, **21**, 3351.**Pentantriol-1 : 2 : 5** $\text{C}_5\text{H}_{12}\text{O}_3$  MW, 120

Liq. B.p. 190–1°/13 mm.  $D_{15}^{20}$  1.136.  $n_D^{20}$  1.42799.

1 : 5-*Di-Me ether* :  $C_7H_{16}O_3$ . MW, 148. B.p. 94–5°/13 mm.  $D_{15}^{20}$  0.976.  $n_D^{14}$  1.43336. p-*Nitrobenzoyl* : m.p. 194–5°.

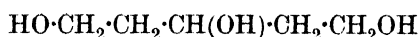
5-*Me 1-Et ether* :  $C_8H_{18}O_3$ . MW, 162. B.p. 99°/13 mm.  $D_4^{17}$  0.955.

*Triacetyl* : b.p. 173–6°/20 mm., 159°/9 mm.  $D_{15}^{15}$  1.123,  $D_{15}^{22}$  1.112.  $n_D^{15}$  1.4402,  $n_D^{22}$  1.4369.

*Triphenylurethane* : m.p. 92°.

Paul, *Ann. chim.*, 1932, **18**, 303; *Bull. soc. chim.*, 1933, **53**, 417.

**Pentantriol-1 : 3 : 5** (1 : 3 : 5-*Trihydroxypentane*)

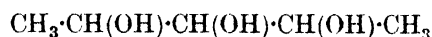


$C_5H_{12}O_3$  MW, 120

Liq. B.p. 188–9°/11 mm.

Blanchard, Paul, *Compt. rend.*, 1935, **200**, 1414.

**Pentantriol-2 : 3 : 4** (1 : 3-*Dimethylglycerol*, 2 : 3 : 4-*trihydroxypentane*)



$C_5H_{12}O_3$  MW, 120

B.p. 244–6°, 152–3°/19 mm. Sol. EtOH,  $H_2O$ . Bitter taste.

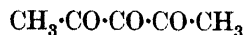
*Triformyl* : m.p. 85°. B.p. 130–4°/15 mm.

*Triacetyl* : needles from  $Et_2O$ . M.p. 121°. B.p. 241–3°.

Delaby, Morel, *Bull. soc. chim.*, 1926, **39**, 416.

Reif, *Ber.*, 1908, **41**, 2740.

**Pentantrione-2 : 3 : 4** (2 : 3 : 4-*Triketopentane*)



$C_5H_6O_3$  MW, 114

Orange-red oil. B.p. 65–70°/30 mm. Bitter taste. Absorbs  $H_2O$  from the air with formation of hydrate, m.p. 52°. Turns brown on standing. Reduces boiling  $CuSO_4$ . Aq. to Cu.

2-*Oxime* : needles from  $AcOEt$ -ligroin. M.p. 75°. Sol.  $H_2O$ , EtOH,  $AcOEt$ . Insol. ligroin.

2 : 3-*Dioxime* : plates from  $H_2O$ . M.p. 128° decomp. Sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ ,  $CHCl_3$ . Conc.  $H_2SO_4$  → green sol.

*Semicarbazone* : leaflets. M.p. 249°. Sol.  $AcOH$ . Spar sol.  $H_2O$ , EtOH, pet. ether. Alkalis → yellow sols.

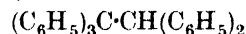
*Phenylhydrazone* : pale yellow leaflets. M.p. 249°. Sol.  $AcOH$ ,  $Me_2CO$ ,  $C_6H_6$ . Insol. pet. ether. Conc.  $H_2SO_4$  → yellow sol.

*Di-phenylhydrazone* : needles. M.p. 156°.

Wolff, *Ann.*, 1902, **325**, 139.

Sachs, Barschall, *Ber.*, 1901, **34**, 3052.

**Pentaphenylethane**



$C_{32}H_{26}$  MW, 410

Plates from pet. ether. M.p. 166–78° in air, 182–5° in nitrogen. Sol.  $CS_2$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ . Very spar. sol. EtOH, pet. ether.  $CrO_3$  → triphenylcarbinol + benzophenone.

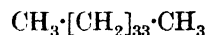
Gomberg, Cone, *Ber.*, 1906, **39**, 1467.

Bachmann, *J. Am. Chem. Soc.*, 1933, **55**, 2135.

**Pentaspadonic Acid.**

See Pelandjaucic Acid.

**Pentatriacontane**



$C_{35}H_{72}$  MW, 492

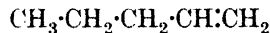
Cryst. from warm EtOH. M.p. 75.0°. B.p. 331°/15 mm.  $D_4^{27}$  0.7816. Spar. sol. boiling  $Et_2O$ .

Easterfield, Taylor, *J. Chem. Soc.*, 1911, **99**, 2305.

Clemmensen, *Ber.*, 1913, **46**, 1842.

Grün, Ulbrich, Krczil, *Z. angew. Chem.*, 1926, **39**, 421.

**1-Pentene** (*Propylethylene*,  $\alpha$ -*amylene*, 1-*pentylene*)



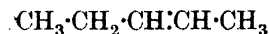
$C_5H_{10}$  MW, 70

B.p. 39° (29.0–29.8°).  $D_4^{20}$  0.6563.  $n_D^{20}$  1.3711. HI → 2-iodopentane.

Adams, Kamm, Marvel, *J. Am. Chem. Soc.*, 1918, **40**, 1950.

Leendertse, Tulleners, Waterman, *Rec. trav. chim.*, 1934, **53**, 715.

**2-Pentene** (sym.-*Methylethylethylene*,  $\beta$ -*amylene*, 2-*pentylene*)



$C_5H_{10}$  MW, 70

F.p. –137°. B.p. 36.39°/760 mm. (36°/741 mm.).  $D_4^{20}$  0.651,  $D_4^{25}$  1.6437.  $n_D^{17}$  1.3817,  $n_D^{20}$  1.3308. HI → 2-iodopentane.

See last reference above and also

Norris, Reuter, *J. Am. Chem. Soc.*, 1927, **49**, 2624.

Norris, *Organic Syntheses*, Collective Vol. I, 421.

**Pentene-carboxylic Acid.**

See 2-Allylpropionic Acid, 3-Ethylidenebutyric Acid, Hydrosorbic Acid, 1-Methyl-2-



ethylacrylic Acid, 1-Methyl-2-ethylidenepropionic Acid, and Propylacrylic Acid.

**Pentene-dial.**

See Glutacondialdehyde.

**1-Pentene-1 : 2-dicarboxylic Acid.**

See Propylfumaric Acid and Propylmaleic Acid.

**1-Pentene-1 : 3-dicarboxylic Acid.**

See 1-Ethylglutaconic Acid.

**2-Pentene-1 : 2-dicarboxylic Acid.**

See Propylidene-succinic Acid.

**2-Pentene-2 : 3-dicarboxylic Acid.**

See dibasic-Hæmatinic Acid.

**2-Pentene-3 : 4-dicarboxylic Acid.**

See 1-Methyl-2-ethylidenesuccinic Acid.

**2-Pentene-3 : 5-dicarboxylic Acid.**

See 1-Ethylideneglutaric Acid.

**2-Pentene oxide.**

See sym.-Methylethylethylene oxide.

**2-Pentene-2 : 3 : 5-tricarboxylic Acid.**

See tribasic-Hæmatinic Acid.

**Pentenic Acid.**

See 2-Ethylidenepropionic Acid, 2-Ethylacrylic Acid, and Allylacetic Acid.

**Pentenol.**

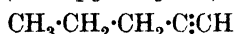
See Allylethyl Alcohol, Ethylvinylcarbinol, Methylallylcarbinol, Methylpropenylcarbinol, and 2-Propylidene-ethyl Alcohol.

**Pentenone.**

See Ethylideneacetone, Ethyl vinyl Ketone, and Methyl allyl Ketone.

**Penthrit.**

See under Pentaerythritol.

**1-Pentine (n-Propylacetylene)**

$\text{C}_5\text{H}_8$

MW, 68

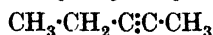
Liq. F.p.  $-95^\circ$ . B.p.  $40^\circ$  ( $48-9^\circ$ ).  $D_{20}^{17}$  0.694.  $n_D^{17}$  1.388.

Picon, *Compt. rend.*, 1914, 158, 1346.

Bouis, *Ann. chim.*, 1928, 9, 402.

Bourguet, *Ann. chim.*, 1925, 3, 191.

Morehouse, Maass, *Chem. Abstracts*, 1935, 29, 1059.

**2-Pentine (Methylethylacetylene)**

$\text{C}_5\text{H}_8$

MW, 68

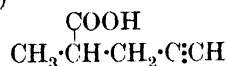
Found in petroleum and fusel oil. F.p.  $-101^\circ$ . B.p.  $55.5^\circ/760$  mm.  $D_{20}^{17.2}$  0.7127.  $n_D^{17.2}$  1.4045.

Faworsky, *J. prakt. Chem.*, 1888, 37, 387.

v. Risseghem, *Compt. rend.*, 1914, 158, 1696.

**1-Pentine-1-carboxylic Acid.**

See Propylpropionic Acid.

**1-Pentine-4-carboxylic Acid (2-Acetylenyl-isobutyric acid)**

$\text{C}_6\text{H}_8\text{O}_2$

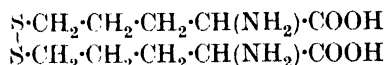
MW, 112

Oil. B.p.  $207-8^\circ/768$  mm. Spar. sol.  $\text{H}_2\text{O}$ .

Et ester:  $\text{C}_8\text{H}_{12}\text{O}_2$ . MW, 140. Oil. B.p.  $165-7^\circ/757$  mm.  $D_{20}^{21}$  0.95424,  $D_{20}^{20}$  0.93989.  $n_D^{17}$  1.42654.

Perkin, Simonsen, *J. Chem. Soc.*, 1907, 91, 832.

Gardner, Perkin, *ibid.*, 853.

**Pentocystine (4 : 4'-Diamino-4 : 4'-dicarboxy-1 : 1'-dibutyl disulphide)**

$\text{C}_{10}\text{H}_{20}\text{O}_4\text{N}_2\text{S}_2$

MW, 296

Needles from  $\text{H}_2\text{O}$ . Decomp. at  $269-72^\circ$ .

Di-formyl: cryst. from  $\text{H}_2\text{O}$ . M.p.  $120-2^\circ$ .

du Vigneaud, Dyer, Jones, Patterson, *J. Biol. Chem.*, 1934, 106, 403.

**Pentylene.**

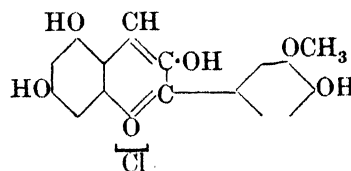
See Pentene.

 **$\gamma$ -Pentylene Glycol.**

See 1 : 4-Dihydroxypentane.

 **$\gamma$ -Pentylene oxide.**

See 2-Methyltetrahydrofuran.

**Peonidin chloride (3 : 5 : 7 : 4'-Tetrahydroxy-3'-methoxyflavylum chloride)**

$\text{C}_{16}\text{H}_{13}\text{O}_6\text{Cl}$

MW, 336.5

Reddish-brown needles +  $\text{H}_2\text{O}$  from 20% HCl. Two modifications of a hydrate containing  $1\frac{1}{2}\text{H}_2\text{O}$  have also been obtained. Sol. EtOH with violet-red col. Mod. sol. cold  $\text{H}_2\text{O}$  with brownish-red col. Spar. sol. dil. HCl.

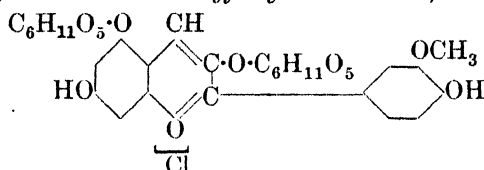
5-Benzoyl: scarlet needles +  $\frac{1}{2}\text{H}_2\text{O}$ . Sol. MeOH, EtOH.

Nolan, Pratt, Robinson, *J. Chem. Soc.*, 1926, 1968.

Murakami, Robinson, *J. Chem. Soc.*, 1928, 1537.

Willstätter, Nolan, *Ann.*, 1915, 408, 141.

**Peonin chloride** (7 : 4'-Dihydroxy-3 : 5-di- $\beta$ -glucosido-3'-methoxyflavylum chloride)



$C_{28}H_{33}O_{16}Cl$

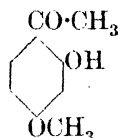
MW, 660.5

Colouring matter of deep violet-red peonies. Reddish-violet needles +  $H_2O$  from aq. HCl. Decomp. at  $165-7^\circ$ . Hyd.  $\rightarrow$  2 mols. glucose + peonidin chloride.

Robinson, Todd, *J. Chem. Soc.*, 1932, 2493.

Willstätter, Nolan, *Ann.*, 1915, 408, 136.

**Peonol** (2-Hydroxy-4-methoxyacetophenone, resacetophenone 4-methyl ether, pæonol)



$C_9H_{10}O_3$

MW, 166

Obtained from the root of *Paeonia Moutan* (China, Japan) and a constituent of several ethereal oils e.g. from *Paeonia arborea*, Don. Needles from EtOH. M.p.  $50^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Volatile in steam.  $D^{20}_D$  1.1310.  $n^{20}_D$  1.54322. Gives reddish-violet col. with  $FeCl_3$  in aq. or alc. solution. KOH fusion  $\rightarrow$  resacetophenone, 2 : 4-dihydroxybenzoic acid, and resorcinol. HI  $\rightarrow$  resacetophenone.  $Ac_2O \rightarrow$  resacetophenone-4-methyl ether acetate. Br in AcOH  $\rightarrow$  bromopeonol.

*Me ether* : 2 : 4-dimethoxyacetophenone. See under Resacetophenone.

*Et ether* : see under Resacetophenone.

*Acetyl* : needles from EtOH. M.p.  $46.5^\circ$ .

*Oxime* : needles. M.p.  $130^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ , ligroin.

*Hydrazone* : plates from dil. EtOH. M.p.  $73-5^\circ$ .

*Phenylhydrazone* : needles from EtOH. M.p.  $108^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH, ligroin. Insol. cold aq. alkalis.

*o-Nitrophenylhydrazone* : deep red prisms from AcOH. M.p.  $217^\circ$ .

*m-Nitrophenylhydrazone* : red plates from AcOH. M.p.  $197^\circ$ .

*p-Nitrophenylhydrazone* : red cryst. from AcOH. M.p.  $238-9^\circ$  ( $235-6^\circ$  decomp.).

2 : 4 : 6-Tribromophenylhydrazone : needles from EtOH. M.p.  $162^\circ$ . Insol. hot aq. alkalis.

Dict. of Org. Comp.—III.

*Semicarbazone* : needles from EtOH. M.p.  $221-2^\circ$ .

Tahara, *Ber.*, 1891, 24, 2460.

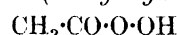
Brüll, Friedländer, *Ber.*, 1897, 30, 300.

Nagai, *Ber.*, 1891, 24, 2847.

Adams, *J. Am. Chem. Soc.*, 1919, 41, 260.

Lindemann, Könitzer, Romanoff, *Ann.*, 1927, 456, 304.

**Peracetic Acid** (*Acetyl hydroperoxide*)



$C_2H_4O_3$

MW, 76

Liq. F.p.  $+0.1^\circ$ . Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $H_2SO_4$ . Explodes violently above  $110^\circ$ . Unpleasant odour. Ketene  $\rightarrow$  diacetyl peroxide. Strong corrosive action on skin. Used as disinfectant.

*Acetyl* : see Diacetyl peroxide.

*Benzoyl* : acetyl benzoyl peroxide. Needles from ligroin. M.p.  $40-1^\circ$ . 0.639 parts sol. in 1000 parts  $H_2O$  at  $25^\circ$ . Stable when pure. Decomp. when damp or in presence of EtOH,  $Et_2O$  or acids. Explodes with hot conc.  $H_2SO_4$ . Pure comp. has no oxidising action.

*m-Nitrobenzoyl* : needles from MeOH-ligroin. M.p.  $68^\circ$ . Explodes on heating.

Konsort f. elektrochem. Ind., D.R.P., 269,937, (*Chem. Zentr.*, 1914, I, 716); 272,738, (*Chem. Zentr.*, 1914, I, 1615).

D'Ans, Frey, *Ber.*, 1912, 45, 1848.

D'Ans, D.R.P., 251,802, (*Chem. Zentr.*, 1912, II, 1413).

Nef, *Ann.*, 1897, 298, 283.

Freer, Nouy, *Am. Chem. J.*, 1902, 27, 172.

**Perbenzoic Acid** (*Benzoyl hydroperoxide*)



$C_7H_6O_3$

MW, 138

Plates from pet. ether. M.p.  $41-3^\circ$ . B.p.  $97-110^\circ/13-15$  mm. Sol. most org. solvents. Spar. sol.  $H_2O$ , pet. ether. Very volatile. Sublimes in exsiccator. Volatile in steam. Strong oxidising agent.

*Acetyl* : see under Peracetic Acid.

*Benzoyl* : see Dibenzoyl peroxide.

Braun, *Organic Syntheses*, 1933, XIII, 86.

Tiffeneau, *Organic Syntheses*, Collective Vol. I, 422.

Hibbert, Burt, *J. Am. Chem. Soc.*, 1925, 47, 2240.

Baeyer, Villiger, *Ber.*, 1900, 33, 858.

v. Pechmann, Vanino, *Ber.*, 1894, 27, 1511.

Gambarjan, *Ber.*, 1909, 42, 4008.

Prileshajew, *Chem. Zentr.*, 1911, I, 1279.

**Perbromoacetone.**

See Hexabromoacetone.

**Perbromobenzoic Acid.**

See Pentabromobenzoic Acid.

**Perbromoethane.**

See Hexabromoethane.

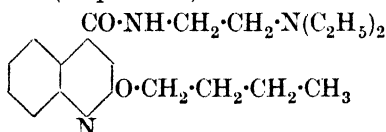
**Perbromoethylene.**

See Tetrabromoethylene.

**Perbromoindone.**

See Hexabromoindone-3.

**Percaïne (Nupercaine)**



$\text{C}_{20}\text{H}_{29}\text{O}_2\text{N}_3$

MW, 343

Plates. M.p. 97–8°. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ . Aq. sol. shows blue fluor. Local anæsthetic about ten times more powerful than cocaine.

Miescher, *Helv. Chim. Acta*, 1932, 15, 169.

Uhlmann, *Chem. Zentr.*, 1929, II, 1816.

**Perchloroacetone.**

See Hexachloroacetone.

**Perchlorobenzene.**

See Hexachlorobenzene.

**Perchlorobenzoic Acid.**

See Pentachlorobenzoic Acid.

**Perchloroethane.**

See Hexachloroethane.

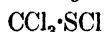
**Perchloroethylene.**

See Tetrachloroethylene.

**Perchloroindone.**

See Hexachloroindone-3.

**Perchloromethyl Mercaptan** (Thiocarbonyl tetrachloride, trichloromethyl sulphochloride)



$\text{CCl}_4\text{S}$

MW, 186

Pale yellow oil. B.p. 149° (146.5–148°), 73°/50 mm.  $D_4^{25}$  1.712,  $D_4^{20}$  1.71785.  $n_D^{20}$  1.54835. Decomp. above 200°.  $\text{SnCl}_2 \rightarrow \text{CSCl}_2 + \text{SnCl}_4$ .  $\text{Fe} + \text{HCl} \rightarrow \text{CSCl}_2 + \text{CCl}_4$ .  $\text{Fe} \rightarrow \text{CCl}_4$ .

Helferich, Reid, *J. Am. Chem. Soc.*, 1921, 43, 591.

Dyson, *Organic Syntheses*, Collective Vol. I, 493.

**Pereirine**

$\text{C}_{20}\text{H}_{26}\text{ON}_2$

MW, 310

Pale yellow amorph. powder +  $\frac{1}{2}\text{H}_2\text{O}$ . M.p. 135° decomp.  $[\alpha]_D + 137.5^\circ$  in EtOH. Sol.

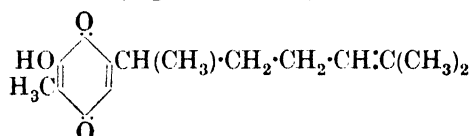
EtOH, MeOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , AcOEt,  $\text{C}_6\text{H}_6$ , acids. Insol.  $\text{H}_2\text{O}$ . Very bitter taste. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  violet-red col.

Me ether:  $\text{C}_{21}\text{H}_{28}\text{ON}_2$ . MW, 324. Yellow amorph. powder. M.p. 195–7° decomp. Very sol. MeOH, EtOH, AcOEt. Insol.  $\text{H}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{Et}_2\text{O}$ , pet. ether.

Methiodide: yellow amorph. powder. M.p. 233–5° decomp. Sol. MeOH, EtOH. Spar. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ .

Bertho, Moog, *Ann.*, 1934, 509, 241.

**Perezone (Pipitzahoic acid)**



$\text{C}_{15}\text{H}_{20}\text{O}_3$

MW, 248

Found in rind of *Trixis pipitzhuac*. Golden-yellow leaflets from  $\text{H}_2\text{O}$ , plates from  $\text{Et}_2\text{O}$ . M.p. 104–6°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , AcOH,  $\text{C}_6\text{H}_6$ . Mod. sol. pet. ether. Insol.  $\text{H}_2\text{O}$ .  $[\alpha]_D^{20} -17.0^\circ$  in  $\text{Et}_2\text{O}$ . Volatile in steam. Racemises readily on sublimation. Alkalis  $\rightarrow$  intense purple col.

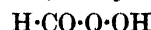
Me ether:  $\text{C}_{16}\text{H}_{22}\text{O}_3$ . MW, 262. B.p. 128–33°/0.002 mm.

Fichter, Jetzer, Leepin, *Ann.*, 1913, 395, 15.

Bayer, D.R.P., 278,090, (*Chem. Zentr.*, 1914, II, 900).

Kögl, Boer, *Rec. trav. chim.*, 1935, 54, 785.

**Performic Acid (Formyl hydroperoxide)**



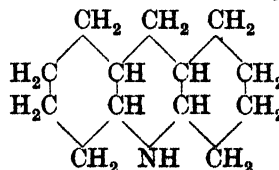
$\text{CH}_2\text{O}_3$

MW, 62

Only obtained 90% pure. Liq. Misc. with  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . More volatile than formic acid. Strong oxidising agent. Explodes on heating with metals and metallic oxides.

D'Ans, Kneips, *Ber.*, 1915, 48, 1137.

**Perhydroacridine (Tetradecahydroacridine)**



$\text{C}_{13}\text{H}_{23}\text{N}$

MW, 193

Cryst. M.p. 80°. B.p. 140°/14 mm.

*B.HCl*: does not melt below 300°. Mod. sol.  $H_2O$ , EtOH.

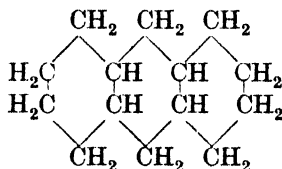
*N-Nitroso*: pale yellow. M.p. 217°.

*Methiodide*: m.p. 266°.

*Picrate*: cryst. M.p. 167°.

v. Braun, Petzold, Schultheiss, *Ber.*, 1923, 56, 1349.

**Perhydroanthracene** (*Tetradecahydroanthracene*)



$C_{14}H_{24}$

MW, 192

Two isomeric compounds have been described.

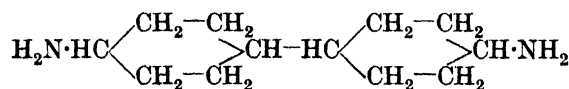
1. Needles from MeOH. M.p. 61.5°.

2. Plates from EtOH. M.p. 93°. B.p. 128°/11 mm.

Fries, Schilling, *Ber.*, 1932, 65, 1494.

Schroeter, *Ber.*, 1924, 57, 1998.

**Perhydrobenzidine** (*Dodecahydrobenzidine*, 4 : 4'-diaminodicyclohexyl)



$C_{12}H_{24}N_2$

MW, 196

Cryst. M.p. 59°. B.p. 120°/0.8 mm. Turns yellowish-brown in air. Odour similar to piperidine. Weak base.

*B.2HCl*: amorph. powder. Decomp. at 290°.

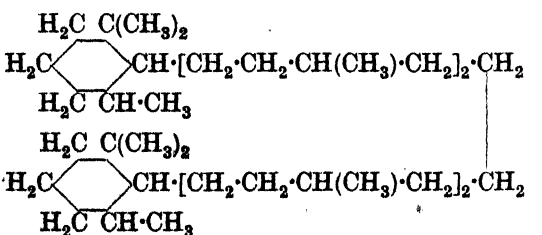
*Dipicrate*: red needles from EtOH. Decomp. explosively at 247°.

Balaš, Ševčenko, *Chem. Zentr.*, 1931, I, 3112.

**Perhydrobixin.**

See Bixane.

**Perhydrocarotene**



$C_{40}H_{78}$

MW, 558

Needles from  $Et_2O$ . M.p. 65°. Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. MeOH, EtOH. Conc.  $H_2SO_4$  → blue sol.

Zechmeister, Cholnoky, Vrabély, *Ber.*, 1928, 61, 566.

**Perhydroindole.**

See Octahydroindole.

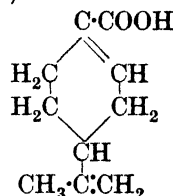
**Peri-Acid.**

See 1-Naphthylamine-8-sulphonic Acid.

**β-Pericyclocamphane.**

See Isobornylene.

**Perillic Acid** (4-Isopropenylcyclohexene-1-carboxylic acid)



$C_{10}H_{14}O_2$

MW, 166

*l.*

Leaflets from EtOH.Aq. M.p. 132-3° (130-1°). B.p. 164-5°/10 mm.

*Amide*:  $C_{10}H_{15}ON$ . MW, 165. Cryst. M.p. 164-5°.

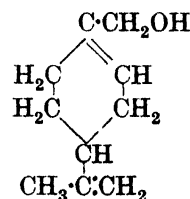
*Nitrile*:  $C_{10}H_{13}N$ . MW, 147. B.p. 123°/15 mm., 116-18°/11 mm.  $D^{20}_D$  0.9488,  $D^{20}_D$  0.9439.  $n^{20}_D$  1.4978.

Schimmel, *Chem. Zentr.*, 1910, II, 1758.

Semmler, Zaar, *Ber.*, 1911, 44, 55.

Furukawa, Tomizawa, *Chem. Abstracts*, 1920, 14, 2839.

**Perillyl Alcohol** ( $\Delta^{1,8(9)}$ -p-Menthadienol-7, 1-hydroxymethyl-4-isopropenylcyclohexene)



$C_{10}H_{16}O$

MW, 152

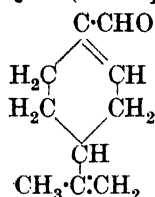
Found in ginger-grass oil. B.p. 118-21°/11 mm., 107-10°/12.5 mm.  $D^{20}_D$  0.9690.  $n_D$  1.4996.  $[\alpha]_D - 7.0^\circ$ .  $CrO_3$  → perillyl aldehyde.

*Acetyl*: b.p. 123-6°/13 mm., 90-1°/4 mm.  $D^{20}_D$  0.9785.  $n^{20}_D$  1.47615.

*Naphthylurethane*: m.p. 146-7°.

See last reference above and also

Semmler, Zaar, *Ber.*, 1911, 44, 54, 460.

**Perillyl Aldehyde** ( $\Delta^{1,8(9)}$ -p-Menthadienal) $C_{10}H_{14}O$ 

MW, 150

l.

Found in oil of *Perilla nankinensis*, D. B.p. 235-7°/750 mm., 104-5°/10 mm., 91°/4.5 mm.  $D_D^{20}$  0.9645.  $n_D^{20}$  1.5069.  $[\alpha]_D^{20}$  -145.8°. Reacts with  $Na_2SO_3$  and  $NaHSO_3$ .  $Ag_2O \rightarrow$  perillic acid.

syn-Oxime : prisms. M.p. 129°.

anti-Oxime : cryst. M.p. 102°. B.p. 147-8°/12 mm. Very sweet.  $B, HCl$  : m.p. 114°.

Semicarbazone : m.p. 199-200°.

Semioxamazone : m.p. 228°.

Phenylhydrazone : needles. M.p. 107.5°.

d.

B.p. 234-6°/743 mm., 99-104°/9 mm.  $D_D^{15}$  0.9730.  $n_D^{20}$  1.5080.  $[\alpha]_D^{15}$  +137°. Reacts with  $Na_2SO_3$  and  $NaHSO_3$ .

Oxime : m.p. 102°.

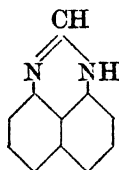
Phenylhydrazone : m.p. 107-8°.

Schimmel, *Chem. Zentr.*, 1910, II, 1758.

Semmler, Zaar, *Ber.*, 1911, 44, 53, 460.

Furukawa, Tomizawa, *Chem. Abstracts*, 1920, 14, 2839.

Hoshino, *ibid.*, 1410.

**Perimidine** (1 : 8-Naphthiminazole, perinaphthiminazole) $C_{11}H_8N_2$ 

MW, 168

Green cryst. from EtOH.Aq. M.p. about 222°. Sol. prac. all org. solvents, acids. Insol.  $H_2O$ .

$B, HCl$  : yellowish-green needles from dil.  $HCl$ . Decomp. at 300°.

$B, HNO_3$  : long green needles from EtOH or hot  $H_2O$ .

Formate : cryst. M.p. 143°, solidifies and then remelts at 220°.

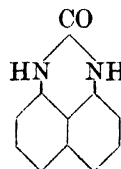
Acetate : cryst. from AcOH.Aq. M.p. 130°, solidifies and then remelts at 221°.

Oxalate : yellow cryst. from hot  $H_2O$  or EtOH. Decomp. at 270°.

Pyruvate : m.p. 207°. Very spar. sol.  $H_2O$ , EtOH.

Picrate : orange-red cryst. from EtOH. M.p. 232° (226°).

Sachs, *Ann.*, 1909, 365, 83.

**Perimidone** $C_{11}H_8ON_2$ 

MW, 184

Cryst. from EtOH or  $C_6H_6$ . M.p. 304-5°. Spar. sol. most org. solvents.

Sachs, *Ann.*, 1909, 365, 135.

**Periodoacetone.**

See Hexaiodoacetone.

**Periodoethylene.**

See Tetraiodoethylene.

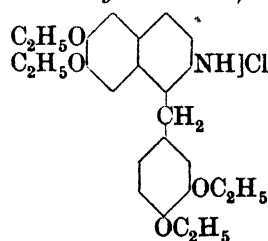
**Perlatolic Acid** $C_{25}H_{32}O_7$ 

MW, 444

Needles from  $C_6H_6$ . M.p. 108°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Spar. sol. pet. ether, ligroin. Sol. alkalis. Alc.  $FeCl_3 \rightarrow$  violet col.  $CHCl_3$  sol. + alkali  $\rightarrow$  red col. on heating showing green fluor. on dilution with  $H_2O$ .

Di-Me ether-Me ester :  $C_{28}H_{38}O_7$ . MW, 486. Needles from MeOH. M.p. 57°.

Asahina, Fuzikawa, *Ber.*, 1935, 68, 634.

**Perparin** (6 : 7-Diethoxy-1-[3' : 4'-diethoxybenzyl]-isoquinoline hydrochloride) $C_{24}H_{30}O_4NCl$ 

MW, 431.5

Cryst. M.p. 99-101°. Used as substitute for papaverine. Possesses stronger pharmacological properties.

Issekutz, Leinzinger, Dirner, *Chem. Abstracts*, 1932, 26, 3836.

Pal, *ibid.*, 2510.

**Perpropionic Acid** (Propionyl hydroperoxide) $CH_3 \cdot CH_2 \cdot CO \cdot O \cdot OH$  $C_3H_6O_3$ 

MW, 90

Liq. F.p.  $-13.5^{\circ}$ . Explodes on heating  $\rightarrow \text{CO}_2 + \text{CH}_4 + \text{C}_2\text{H}_4 + \text{C}_2\text{H}_6$ .

*Propionyl*: dipropionyl peroxide. Liq. Decomp. above  $80^{\circ}$ . Misc. with usual solvents.

Clover, Richmond, *Am. Chem. J.*, 1903, **29**, 191.

Konsort f. elektrochem. Ind., D.R.P., 269,937, (*Chem. Zentr.*, 1914, **I**, 716).

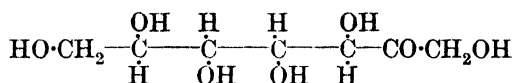
D'Ans, Frey, *Ber.*, 1912, **45**, 1850.

Fichter, Krummenacher, *Helv. Chim. Acta*, 1918, **1**, 146.

### Perseitol.

See Persitol.

### Perseulose



$\text{C}_7\text{H}_{14}\text{O}_7$

MW, 210

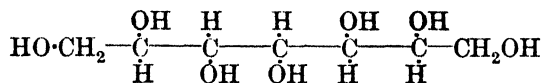
Needles from  $\text{H}_2\text{O}$ . M.p.  $110-15^{\circ}$  decomp. Sol. boiling EtOH. Sweet taste. Aq. sol. shows mutarotation.  $[\alpha]_D^{20} - 90.0^{\circ} \rightarrow - 81.0^{\circ}$  in  $\text{H}_2\text{O}$ . Reduces Fehling's.

*Phenylosazone*: needles from EtOH. M.p.  $233^{\circ}$ .

Bertrand, *Bull. soc. chim.*, 1909, **5**, 629.

La Forge, *J. Biol. Chem.*, 1916, **28**, 514.

### Persitol (*Perseitol*, d- $\alpha$ -mannoheptitol)



$\text{C}_7\text{H}_{16}\text{O}_7$

MW, 212

Obtained from seeds of *Persea gratissima* or *P. dymifolia*. Needles. M.p.  $188^{\circ}$ . Sol.  $\text{H}_2\text{O}$ , hot EtOH. Spar. sol.  $\text{Et}_2\text{O}$ .  $[\alpha]_D^{20} + 4.53^{\circ}$ . Oxidised by *B. xylinum* to perseulose.

*Hepta-acetyl*: m.p.  $119^{\circ}$ . Sol. EtOH. Insol.  $\text{H}_2\text{O}$ .

*Heptanitrate*: needles from EtOH. M.p.  $138^{\circ}$ .

Maquenne, *Ann. chim. phys.*, 1890, **19**, 5.

Fischer, *Ber.*, 1890, **23**, 936.

Bertrand, *Compt. rend.*, 1909, **149**, 226.

La Forge, *J. Biol. Chem.*, 1917, **28**, 520.

### Peruvial.

See Nerolidol.

### Perylene



$\text{C}_{20}\text{H}_{12}$

MW, 252

Bronze plates from toluene or AcOH. M.p.  $273-4^{\circ}$  ( $264-5^{\circ}$ ). Sublimes at  $350-400^{\circ}$  in bronze leaflets. Sol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ . Less sol.  $\text{C}_6\text{H}_6$ . Mod. sol. AcOH. Spar. sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ . Insol. ligroin. Very dil. sols. show blue fluor. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  deep green sol. which rapidly changes to bluish-green  $\rightarrow$  blue  $\rightarrow$  reddish-violet.  $\text{CrO}_3$  in boiling AcOH  $\rightarrow$  perylenequinone.

Scholl, Seer, Weitzenböck, *Ber.*, 1910, **43**, 2203.

Hansging, Zinke, *Monatsh.*, 1919, **40**, 404.

Marschalk, *Bull. soc. chim.*, 1928, **43**, 1388.

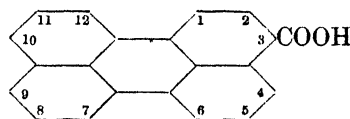
Zinke, Hauswirth, Blank, Grimm, *Monatsh.*, 1932, **61**, 1.

Clar, *Ber.*, 1932, **65**, 846.

Uchida, Takata, *J. Soc. Chem. Ind. Japan*, 1933, **36**, 222.

Morgan, Mitchell, *J. Chem. Soc.*, 1934, 536.

### Perylene-3-carboxylic Acid



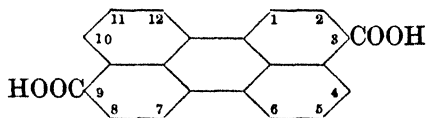
$\text{C}_{21}\text{H}_{12}\text{O}_2$

MW, 296

Yellowish-brown needles from  $\text{PhNO}_2$ . Does not melt below  $300^{\circ}$ . Sol.  $\text{H}_2\text{SO}_4$  with violet col. and red fluor. Alkalis  $\rightarrow$  yellow sols. with bluish-green fluor.

I.G., F.P., 635,599, (*Chem. Zentr.*, 1929, **I**, 2472).

### Perylene-3 : 9-dicarboxylic Acid



$\text{C}_{22}\text{H}_{12}\text{O}_4$

MW, 340

Orange micro-needles from  $\text{PhNO}_2$ . Sinters about  $360^{\circ}$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  red sol. with orange-red fluor. Aq. alkalis  $\rightarrow$  yellow sols. with bluish-green fluor. Spar. sol. org. solvents.

*Di-Et ester*:  $\text{C}_{26}\text{H}_{28}\text{O}_4$ . MW, 396. Yellow leaflets from xylene. M.p.  $264-5^{\circ}$ . Spar. sol. EtOH, toluene, xylene. Sols. are yellow with green fluor.

*Dichloride*:  $\text{C}_{22}\text{H}_{10}\text{O}_2\text{Cl}_2$ . MW, 377. Red needles from  $\text{PhNO}_2$ . Sol.  $\text{C}_6\text{H}_6$ , toluene, xylene. Sols. are yellow with yellowish-green fluor.

**Dinitrile**:  $C_{22}H_{10}N_2$ . MW, 302. Brown cryst. from  $PhNO_2$ . Does not melt below  $360^\circ$ . Spar. sol. EtOH, AcOH, xylene.

Pongratz, *Monatsh.*, 1927, **48**, 585; 1929, **52**, 9.

Pongratz, Griengl, Cecelsky, *Monatsh.*, 1933, **62**, 71.

### Perylene-3 : 10-dicarboxylic Acid.

Reddish-brown needles from  $PhNO_2$ . Sol. 1000 parts hot  $PhNO_2$ . Conc.  $H_2SO_4 \rightarrow$  reddish-violet col. slowly changing to brownish-red. Aq. alkalis  $\rightarrow$  yellow sols. with strong green fluor.

**Di-Et ester**: red leaflets from xylene. M.p.  $247-8^\circ$ .

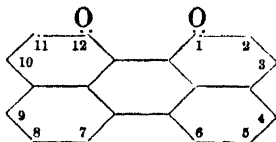
**Dichloride**: reddish brown needles from benzoyl chloride.

**Dinitrile**: brown needles from aniline. M.p.  $368-9^\circ$  (sealed tube). Sol.  $PhNO_2$  aniline. Spar. sol. AcOH, xylene with intense green fluor. Conc.  $H_2SO_4 \rightarrow$  reddish-brown col.

Weitzenböck, Seer, *Ber.*, 1913, **46**, 1999.

Funke, *Monatsh.*, 1932, **59**, 193.

### 1 : 12-Perylenequinone



$C_{20}H_{10}O_2$

MW, 282

Reddish-brown needles from  $C_6H_6$ . M.p.  $287^\circ$ . Sol. hot AcOH, Py,  $PhNO_2$ , aniline, chlorobenzene. Spar. sol. hot EtOH,  $Me_2CO$ ,  $CCl_4$ ,  $C_6H_6$ , toluene. Conc.  $H_2SO_4 \rightarrow$  brown sol. Insol. NaOH.

Zinke, Hanselmayer, *Monatsh.*, 1924, **45**, 232.

### 3 : 9-Perylenequinone.

Violet needles from  $PhNO_2$ . Reduced by hydrosulphite to red "vat" with intense green fluor.

Zinke, Hirsch, *Monatsh.*, 1929, **52**, 18.

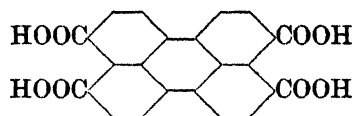
### 3 : 10-Perylenequinone.

Yellow needles from Py. Decomp. slowly above  $350^\circ$ . Sol. boiling AcOH, Py,  $PhNO_2$ , aniline, xylene, quinoline. Conc.  $H_2SO_4 \rightarrow$  blood-red sol. with intense fluor.

Zincke, Unterkreuter, *Monatsh.*, 1919, **40**, 407.

Marschalk, *Bull. soc. chim.*, 1927, **41**, 74.

### Perylene - 3 : 4 : 9 : 10 - tetracarboxylic Acid



$C_{24}H_{12}O_8$

MW, 428

Brownish-red needles. Hot alkalis  $\rightarrow$  brownish-yellow sols. with deep green fluor. Conc.  $H_2SO_4 \rightarrow$  red sol. with orange-red fluor. Dry dist. of Ca salt  $\rightarrow$  perylene.

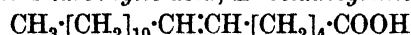
**Monimide**: brownish-red powder. Very dil. alkalis  $\rightarrow$  deep red sols.

Kalle, D.R.P., 394,794, (*Chem. Zentr.*, 1924, II, 1276); 408,513, (*Chem. Zentr.*, 1925, I, 1811); 412,122, (*Chem. Zentr.*, 1925, I, 2666).

### Petroselaidic Acid.

See under Petroselic Acid.

**Petroselic Acid** (Petroselinic acid, 5-hepta-decylene-1-carboxylic acid,  $\Delta^5$ -octadecylenic acid)



$C_{18}H_{34}O_2$

MW, 282

**Cis**:

Found in seeds of *Petroselinum sativum*. Leaflets. F.p.  $27^\circ$ . M.p.  $33-4^\circ$  ( $30^\circ$ ). B.p.  $237-8^\circ/18$  mm.  $D_{20}^{40}$  0.8700.  $n_D^{40}$  1.4533. Dil.  $HNO_3 \rightarrow$  *trans* acid. Ozone  $\rightarrow$  adipic aldehyde + lauric acid.

**Amide**:  $C_{18}H_{35}ON$ . MW, 281. Needles. M.p.  $76^\circ$ .

**Triglyceride**: tripetroselin. Cryst. F.p.  $16.5^\circ$ . M.p.  $32^\circ$  ( $26.4^\circ$ ).  $n_D^{40}$  1.4619.

**Trans**: Petroselaidic acid.

Prisms. M.p.  $54^\circ$ .

Palazzo, Tamburello, *Atti accad. Lincei*, 1914, **23**, II, 352.

Vongerichten, Köhler, *Ber.*, 1909, **42**, 1638.

Steger, Loon, *Rec. trav. chim.*, 1927, **46**, 703.

Hilditch, Jones, *J. Soc. Chem. Ind.*, 1927, **46**, 174T.

Eibner, Widenmeyer, Schild, *Chem. Abstracts*, 1928, **22**, 1487.

### Petroselinic Acid.

See Petroselic Acid.

### Petunidin (chloride)

$C_{18}H_{19}O_7Cl$

MW, 352.5

Forms several hydrates of different cryst. forms and solubilities depending on solvent used. Very similar in properties to myrtillidin chloride.

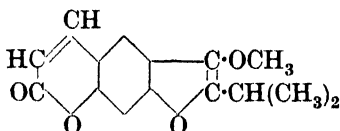
Willstätter, Burdick, *Ann.*, 1916, **412**, 224.

Cf. Bell, Robinson, *J. Chem. Soc.*, 1934, 1604.

**Petunin (chloride)** $C_{28}H_{33}O_{17}Cl$  MW, 676.5

Plates +  $2H_2O$ . M.p. about  $179^\circ$ . Cryst. are violet by transmitted light, and show copper lustre by reflected light. Sol. MeOH. MeOH +  $FeCl_3 \rightarrow$  violet col.  $Na_2CO_3$ . Aq.  $\rightarrow$  violet sol.  $\rightarrow$  blue on standing. 20% HCl  $\rightarrow$  petunidin chloride + dextrose.

Willstätter, Burdick, *Ann.*, 1916, **412**, 217.

**Peucedanine** $C_{15}H_{14}O_4$  MW, 258

Found in roots of *Peucedanum officinale*. Prisms or plates. M.p.  $109^\circ$  ( $76^\circ$ ). Very sol.  $CHCl_3$ ,  $CS_2$ . Sol. hot EtOH,  $Et_2O$ , AcOH. Spar. sol.  $C_6H_6$ , pet. ether. Insol.  $H_2O$ .  $HNO_3 \rightarrow$  styphnic acid. Tasteless. Toxic to fish.

Heut, *Ann.*, 1875, **176**, 71.

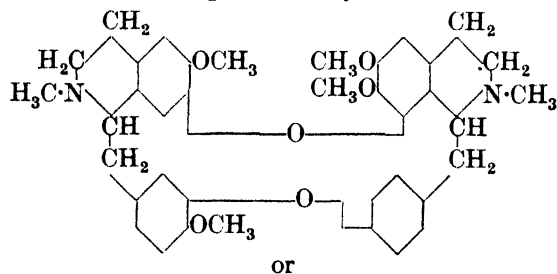
Jassoy, Haensel, *Arch. Pharm.*, 1898, **236**, 668.

Späth, Klager, *Ber.*, 1933, **66**, 749.

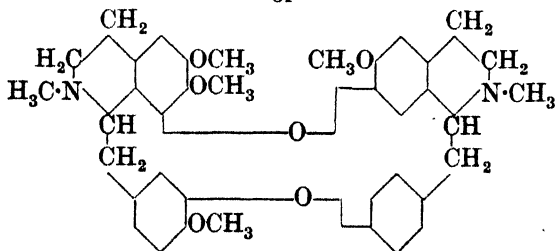
Späth, Klager, Schlösser, *Ber.*, 1931, **64**, 2203.

**Phaeanthine (1-Tetrandrine)**

Constitution represented by



or

 $C_{38}H_{42}O_6N_2$  MW, 622

Alkaloid extracted from *Stephania cepharantha*, *Cocculus lavrifolius*, and *Phaeanthus*

*ebacteolatus*. Hexagonal prisms from  $Et_2O$ . M.p.  $210^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ . Mod. sol. MeOH,  $Me_2CO$ . Spar. sol. EtOH.  $[\alpha]_D^{30} - 278^\circ$  in  $CHCl_3$ .

$B,2HI$ : cryst. Decomp. at  $268^\circ$ .

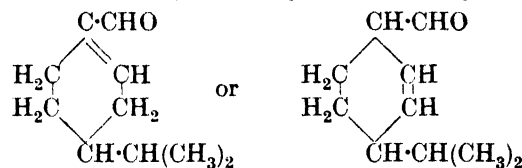
$B,2CH_3I$ : decomp. at  $265^\circ$ .

*Picrate*: decomp. at  $263^\circ$ .

Kondo, Keimatsu, *Ber.*, 1935, **68**, 1505.

**Phaseolunatin.**

See Linamarin.

**Phellandral ( $\Delta^1$ -Tetrahydrocuminaldehyde)** $C_{10}H_{16}O$  MW, 152

Occurs in water-fennel oil. B.p.  $220-30^\circ$ ,  $89^\circ/5$  mm. Mod. sol.  $H_2O$ .  $D_D^{20}$  0.93.  $n_D^{20}$  1.4911 (1.4903). Ox.  $\rightarrow$  tetrahydrocuminic acid.

*Oxime*: m.p.  $87-8^\circ$ .

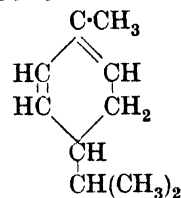
*Semicarbazone*: m.p.  $204-5^\circ$ .

*p*-Nitrophenylhydrazone: m.p.  $169-70^\circ$ .

2:4-Dinitrophenylhydrazone: orange-red needles from  $CHCl_3$ -EtOH. M.p.  $202-3^\circ$ .

Macbeth, Price, *J. Chem. Soc.*, 1935, 152.

Wallach, *Ann.*, 1905, **340**, 13.

 **$\alpha$ -Phellandrene ( $\Delta^{1,5}$ -p-Menthadiene, 1-methyl-4-isopropylcyclohexadiene-1:5)** $C_{10}H_{16}$  MW, 136

*d.*

Occurs in bitter-fennel, elemi, and ginger-grass oils. B.p.  $175-6^\circ$ ,  $66-8^\circ/16$  mm.,  $66^\circ/14$  mm.,  $61^\circ/11$  mm.  $D_D^{20}$  0.8463.  $n_D^{20}$  1.4777.

$\alpha$ -Nitrosite: m.p.  $113-14^\circ$  (rapid heat).  $[\alpha]_D^{20} - 141^\circ$  in AcOEt.

$\beta$ -Nitrosite: m.p.  $105^\circ$ .  $[\alpha]_D^{19} + 45.8^\circ$  in  $CHCl_3$ .

*l.*

Occurs in oils of pimento, bay, and *Eucalyptus phellandra*, Smith. B.p.  $174-7^\circ/759$  mm.,  $171-2^\circ/758.2$  mm.,  $67-8^\circ/22$  mm.,  $58-9^\circ/16$  mm.



(60–60.5°/15 mm.).  $D_4^{20}$  0.8425.  $n_D^{20}$  1.4732.  $[\alpha]_D^{20} - 112.76^\circ$ .

$\alpha$ -Nitrosite : m.p. 120–1°.

$\beta$ -Nitrosite : m.p. 105–6°.

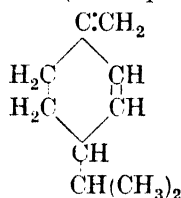
$d_L$ .

B.p. 175–6° decomp., 63–5°/15.5 mm.  $n_D^{19.5}$  1.4772.

Read, Storey, *J. Chem. Soc.*, 1930, 2781.

Galloway, Dewar, Read, *J. Chem. Soc.*, 1936, 1597.

**$\beta$ -Phellandrene** ( $\Delta^{2,1(7)}$ -p-Menthadiene)



$C_{10}H_{16}$

MW, 136

$d_L$ .

Occurs in oil of *Bupleurum fruticosum*, Linn. B.p. 171–2°/766 mm., 57°/11 mm.  $D^{20}$  0.8520.  $n_D^{20}$  1.4788.

$\alpha$ -Nitrosochloride : m.p. 101–2°.  $[\alpha]_D - 175^\circ$  in  $CHCl_3$ .

$\beta$ -Nitrosochloride : m.p. 100–1°.  $[\alpha]_D - 285^\circ$  in  $CHCl_3$ .

$\alpha$ -Nitrosite : m.p. 102°.  $[\alpha]_D^{18.5} - 159.3^\circ$  in  $CHCl_3$ .

$\beta$ -Nitrosite : m.p. 97–8°.  $[\alpha]_D - 159^\circ$  in  $CHCl_3$ .

Francesconi, Sernagiotto, *Gazz. chim. ital.*, 1916, **46**, i, 119.

Wallach, *Ann.*, 1905, **340**, 1.

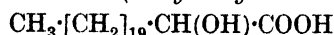
**$\alpha$ -Phellandrene Hydrate.**

See  $\Delta^2$ -p-Menthanol-1.

**Phellogenic Acid.**

See Japanic Acid.

**Phellonic Acid** (1-Hydroxybehenic acid)



$C_{22}H_{44}O_3$

MW, 356

Cryst. from  $CHCl_3$ . M.p. 96°.

*Et ester* :  $C_{24}H_{48}O_3$ . MW, 384. M.p. 70–1°.

*Acetyl* : m.p. 79°.

*Et ether* :  $C_{24}H_{48}O_3$ . MW, 384. M.p. 60°.

Fileti, *Gazz. chim. ital.*, 1897, **27**, ii, 300.

Zetzsche, Bähler, *Helv. Chim. Acta*, 1931, **14**, 642.

**Phenacaine.**

See under Holocaine.

**Phenacetin.**

See under p-Phenetidine.

**Phenaceturic Acid** (Phenylacetylglutamine)



$C_{10}H_{11}O_3N$

MW, 193

Occurs in horse and dog urine. Leaflets from EtOH. M.p. 143° (136°). Sol. EtOH. Spar. sol.  $H_2O$ ,  $Et_2O$ , hot  $C_6H_6$ . Hot  $HCl \rightarrow$  glycine + phenylacetic acid.

*Me ester* :  $C_{11}H_{13}O_3N$ . MW, 207. Cryst. from EtOH. M.p. 86.5°. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , hot EtOH.

*Et ester* :  $C_{12}H_{15}O_3N$ . MW, 221. Prisms from EtOH. M.p. 82° (79°). Sol. hot EtOH.

*Propyl ester* :  $C_{13}H_{17}O_3N$ . MW, 235. Leaflets from  $H_2O$ . M.p. 31°.

*Amide* :  $C_{10}H_{12}O_2N_2$ . MW, 192. Plates from EtOH.Aq. M.p. 176–7°. Sol. hot  $H_2O$ . Insol. EtOH,  $Et_2O$ .

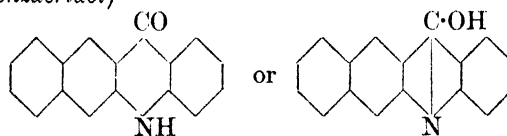
*Nitrile* :  $C_{10}H_{10}ON_2$ . MW, 174. Needles. M.p. 90.5°. Spar. sol. hot  $H_2O$ .

Hotter, *J. prakt. Chem.*, 1888, **38**, 98.

**Phenacridol.**

See Phenacridone.

**Phenacridone** (Phenacridol, benzacridone, benzacridol)



$C_{17}H_{11}ON$

MW, 245

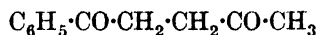
Yellow leaflets from AcOH. M.p. 304–5°. Spar. sol. ord. org. solvents. Sols. show green fluor.

*N-Et* :  $C_{19}H_{15}ON$ . MW, 273. Needles from EtOH. M.p. 174–5°.

*N-Benzyl* :  $C_{24}H_{17}ON$ . MW, 335. Yellow needles from EtOH. M.p. 188–9°.

Schöpf, *Ber.*, 1893, **26**, 2590.

**Phenacylacetone** (Acetophenoneacetone, 1-acetyl-2-benzoylthane,  $\alpha$  : 8-diketo-n-amylbenzene,  $\omega$ -acetonylacetophenone)



$C_{11}H_{12}O_2$

MW, 176

Yellow oil. B.p. 161°/12 mm. Sol. hot  $H_2O$ . Insol. alkalis. No col. with  $FeCl_3$ .

*Monoxime* : m.p. 123°.

*Dioxime* : m.p. 108°.

*Mono-semicarbazone* : m.p. 191°.

Borsche, Fels, *Ber.*, 1906, **39**, 1926.

See also Finzi, *Gazz. chim. ital.*, 1912, **42**, ii, 356.

**Phenacyl Alcohol** (*Acetophenone alcohol benzoylcarbinol, ω-hydroxyacetophenone*)


 $\text{C}_8\text{H}_8\text{O}_2$ 

MW, 136

Prisms from ligroin, plates from EtOH. M.p. anhyd. 86–7° (73–4° hydrated). B.p. 118–20°/11 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. ligroin, hot H<sub>2</sub>O. Sublimes at 56°/1 mm.

*Me ether*: ω-methoxyacetophenone. C<sub>9</sub>H<sub>10</sub>O<sub>2</sub>. MW, 150. M.p. 7–7.5°. B.p. 228–30°, 118–20°/15 mm. *Semicarbazone*: m.p. 129°. 2:4-*Dinitrophenylhydrazones*: m.p. 191–2°.

*Et ether*: C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>. MW, 164. M.p. 81°. B.p. 134–6°/21 mm. *Oxime*: m.p. 55°.

*Phenyl ether*: C<sub>14</sub>H<sub>12</sub>O<sub>2</sub>. MW, 212. M.p. 78°. B.p. 255–7°. Volatile in steam. *Oxime*: m.p. 113–14°.

*o*-Nitrophenyl ether: m.p. 118°.

*p*-Nitrophenyl ether: m.p. 144°.

*o*-Tolyl ether: m.p. 84°.

*p*-Tolyl ether: m.p. 68°.

*Oxime*: m.p. 70°.

*Acetyl*: m.p. 49° (40°). B.p. 270°.

*Benzoyl*: m.p. 118.5°. *Oxime*: m.p. 92°.

*o*-Nitrobenzoyl: m.p. 124.5°.

*m*-Nitrobenzoyl: m.p. 104.5°.

*p*-Nitrobenzoyl: m.p. 128.4°.

Rather, Reid, *J. Am. Chem. Soc.*, 1919, **41**, 79.

Wolff, *Ann.*, 1912, **394**, 42.

Dakin, *J. Biol. Chem.*, 1914, **18**, 92.

Allen, Scarrow, *Chem. Zentr.*, 1935, **I**, 1870.

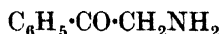
Hann, Reid, Jamieson, *J. Am. Chem. Soc.*, 1930, **52**, 818.

Kelly, Kleff, *J. Am. Chem. Soc.*, 1932, **54**, 4444.

### Phenacyl Aldehyde.

See ω-Formylacetophenone.

**Phenacylamine** (*Phenomydrol, ω-aminoacetophenone, benzoylmethylamine*)


 $\text{C}_8\text{H}_9\text{ON}$ 

MW, 135

*B, HCl*: m.p. 186–7° decomp. (183–4°).

*B, HBr*: m.p. 217–18° decomp.

*Sulphate*: m.p. 182°.

*N-Benzoyl*: m.p. 126°.

*N-p-Toluenesulphonyl*: m.p. 79° decomp.

*Oxime*: m.p. 140°.

*Picrate*: m.p. 175–6° decomp.

Kindler, D.R.P., 571,795, (*Chem. Abstracts*, 1933, **27**, 4245).

Neber, Huh, *Ann.*, 1935, **515**, 292.

Neber, F.P., 768,604, (*Chem. Abstracts*, 1935, **29**, 475).

Tiffeneau, Orekoff, Roger, *Bull. soc. chim.*, 1931, **49**, 1761.

### Phenacyl-o-aminobenzoic Acid.

See Phenacylanthranilic Acid.

### Phenacyl-m-aminobenzoic Acid

COOH


 $\text{C}_{15}\text{H}_{13}\text{O}_3\text{N}$ 

MW, 255

Cryst. from EtOH. M.p. 202°. Sol. AcOH, Py.

*N-Acetyl*: m.p. 217°.

Scholtz, *Ber.*, 1918, **51**, 1652.

### Phenacyl-p-aminobenzoic Acid.

Needles from EtOH. M.p. 211°.

*Phenacyl ester*: C<sub>23</sub>H<sub>19</sub>O<sub>4</sub>N. MW, 373. M.p. 186°.

*N-Acetyl*: m.p. 176°.

Scholtz, *Ber.*, 1918, **51**, 1653.

### Phenacylaniline.

See ω-Anilinoacetophenone.

**Phenacylanthranilic Acid** (*Phenacyl-o-aminobenzoic acid*)

COOH


 $\text{C}_{15}\text{H}_{13}\text{O}_3\text{N}$ 

MW, 255

Yellow needles from EtOH.Aq. M.p. 190°. Sol. ord. org. solvents.

*Phenacyl ester*: C<sub>23</sub>H<sub>19</sub>O<sub>4</sub>N. MW, 373. M.p. 180°.

*Phenylhydrazones*: m.p. 156°.

Scholtz, *Ber.*, 1918, **51**, 1648.

**o-Phenacylbenzoic Acid** (*Deoxybenzoin-2-carboxylic acid, phenyl o-carboxybenzyl ketone*)

COOH


 $\text{C}_{15}\text{H}_{12}\text{O}_3$ 

MW, 240

Needles from EtOH.Aq. M.p. 169–70° (162–3°).

**Methylamide**:  $C_{18}H_{15}O_2N$ . MW, 253. M.p. 143–4°.

Auwers, Auffenberg, *Ber.*, 1919, **52**, 109.  
Graebe, Trümper, *Ber.*, 1898, **31**, 377.

**Phenacyl bromide** ( $\omega$ -Bromoacetophenone, bromomethyl phenyl ketone)

$C_8H_7OBr$   $C_6H_5 \cdot CO \cdot CH_2Br$  MW, 199

Prisms from EtOH.Aq. M.p. 51° (50°). B.p. 135°/18 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. D<sup>15</sup> 1.709.  $KMnO_4 \rightarrow$  benzoic acid.

**Oxime**: (i) m.p. 89.5°. **Acetyl deriv.**: m.p. 145°. (ii) M.p. 96.5–97°.

**Semicarbazone**: m.p. 146°.

Clibbens, Nierenstein, *J. Chem. Soc.*, 1915, **107**, 1492.

Rather, Reed, *J. Am. Chem. Soc.*, 1919, **41**, 77.

**Phenacylcarbinol.**

See  $\gamma$ -Hydroxypropiofenone.

**Phenacyl chloride** ( $\omega$ -Chloroacetophenone, chloromethyl phenyl ketone)

$C_8H_7OCl$   $C_6H_5 \cdot CO \cdot CH_2Cl$  MW, 154.5

Leaflets from pet. ether. M.p. 58–9°. B.p. 244–5°, 139–41°/14 mm. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. D<sup>15</sup> 1.324.  $CrO_3 \rightarrow$  benzoic acid.

**Oxime**: m.p. 88.5–89°. **Acetyl deriv.**: m.p. 67–9°.

**Semicarbazone**: m.p. 156° (149°, 137°).

Clibbens, Nierenstein, *J. Chem. Soc.*, 1915, **107**, 1492.

Béhal, Detoeuf, *Compt. rend.*, 1911, **153**, 1231.

**$\omega$ -Phenacylcresol.**

See  $\gamma$ -Hydroxyphenylpropiofenone.

**Phenacylformaldehyde.**

See  $\omega$ -Formylacetophenone.

**Phenacylglycollic Acid** (2-Benzoyl-lactic acid, 1-hydroxy-3-keto-3-phenyl-n-butyric acid)

$C_6H_5 \cdot CO \cdot CH_2 \cdot CH(OH) \cdot COOH$   
 $C_{10}H_{10}O_4$  MW, 194

Cryst. M.p. 127° (125–6°). Sol. hot H<sub>2</sub>O. Insol. ligroin. Hot alkalis  $\rightarrow$  oxalic acid + acetophenone.

Kozniewski, Marchlewski, *Chem. Zentr.*, 1906, II, 1190.

Koenigs, Wagstaffe, *Ber.*, 1893, **26**, 557.

**Phenacylgyoxylic Acid.**

See Benzoylpyruvic Acid.

**Phenacylhydrazine** ( $\omega$ -Hydrazinoacetophenone)

$C_6H_5 \cdot CO \cdot CH_2 \cdot NH \cdot NH_2$   
 $C_8H_{10}ON_2$  MW, 150

Leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 85–6° decomp. Unstable at ord. temps. Sol. hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

**B, (COOH)<sub>2</sub>, H<sub>2</sub>O**: m.p. 149–50° decomp.

**N : N'-Diacetyl**: m.p. 123°.

**$\omega$ -N-o-Nitrobenzylidene**: m.p. 156° decomp.

**$\omega$ -N-m-Nitrobenzylidene**: m.p. 146–7°.

**$\omega$ -N-o-Hydroxybenzylidene**: m.p. 110° decomp.

Busch, Foerst, *J. prakt. Chem.*, 1928, **119**, 287.

**Phenacylhydrocinnamic Acid.**

See 2-Phenyl-3-benzoylbutyric Acid and 2-Phenyl-2'-benzoylisobutyric Acid.

**Phenacylidene bromide** ( $\omega$ -Dibromoacetophenone)

$C_6H_5 \cdot CO \cdot CHBr_2$   
 $C_8H_6OBr_2$  MW, 278

Plates from CHCl<sub>3</sub>. M.p. 35–6° (36–7°). B.p. 175–6°/23 mm.  $KMnO_4 \rightarrow$  benzoic acid.

Evans, Brooks, *J. Am. Chem. Soc.*, 1908, **30**, 406.

Wittorf, *Chem. Zentr.*, 1900, II, 29.

**Phenacylidene chloride** ( $\omega$ -Dichloroacetophenone)

$C_6H_5 \cdot CO \cdot CHCl_2$   
 $C_8H_6OCl_2$  MW, 189

M.p. 20–21.5°. B.p. 249°, 143°/25 mm., 131–2°/11 mm.  $KMnO_4 \rightarrow$  benzoic acid.

Jackson, *J. Am. Chem. Soc.*, 1934, **56**, 977.

Gautier, *Ann. chim.*, 1888, **14**, 345, 385.

See also last reference above.

**Phenacylidene iodide** ( $\omega$ -Di-iodoacetophenone)

$C_6H_5 \cdot CO \cdot CHI_2$   
 $C_8H_6OI_2$  MW, 372

Oil. B.p. above 200° decomp. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Insol. H<sub>2</sub>O.

Collet, *Bull. soc. chim.*, 1900, **23**, 830.

Wolff, *Ann.*, 1902, **325**, 143.

**2-Phenacylidenemethylfuran.**

See Furfurylideneacetophenone.

**Phenacyl iodide** ( $\omega$ -Iodoacetophenone, iodo-methyl phenyl ketone)

$C_8H_7OI$   $C_6H_5 \cdot CO \cdot CH_2I$  MW, 246

M.p. 28–30°. B.p. 158–60°/15 mm.

Truchet, *Ann. chim.*, 1931, **16**, 373.

**Phenacyl Mercaptan** ( $\omega$ -Mercaptoacetophenone)

$C_8H_8OS$   $C_6H_5 \cdot CO \cdot CH_2SH$  MW, 152

M.p. 23–4°. B.p. 116–22°/4 mm.  $D_4^{20}$  1.1753. Readily decomp.  $\rightarrow H_2S$ .  $ZnHg + HCl \rightarrow$  ethylbenzene quantitatively.

Oxime : m.p. 70°.

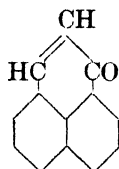
Phenylhydrazone : m.p. 90–1°.

Groth, *Chem. Abstracts*, 1924, **18**, 1281.

**p-Phenacylphenol.**

See 4-Hydroxydeoxybenzoin.

**Phenalone** (peri-Naphthindenone, perinaphthindone)



$C_{13}H_8O$  MW, 180

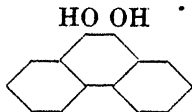
Canary-yellow leaflets from MeOH or cyclohexene. M.p. 153–4°. Basic. Sol. conc. HCl,  $+ H_2O \rightarrow$  red col. Conc.  $H_2SO_4 \rightarrow$  yellow sol. with intense green fluor.

Badische, D.R.P., 283,066, (*Chem. Zentr.*, 1915, I, 814).

Cook, Hewitt, *J. Chem. Soc.*, 1934, 368, 373.

See also Braun, Manz, Reinsch, *Ann.*, 1929, **468**, 301.

**Phenanthrahydroquinone** (9 : 10-Dihydroxyphenanthrene, phenanthraquinol, phenanthrene-9 : 10-diol)



$C_{14}H_{10}O_2$  MW, 210

Needles. M.p. 148°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , hot  $H_2O$ . Heat of comb.  $C_r$  1604.3 Cal. Ox. in air  $\rightarrow$  9 : 10-phenanthraquinone.

Monoacetyl : m.p. 168–70°.

Diacetyl : m.p. 202°.

Dibenzoyl : m.p. 216–17°.

Mono-o-xylyl ether :  $C_{22}H_{18}O_2$ . MW, 314. Yellow cryst. from ligroin. M.p. 148–9°.

Mono-p-xylyl ether : yellow needles from ligroin. M.p. 129–30°.

Braun, Bayer, *Ber.*, 1925, **58**, 2680.

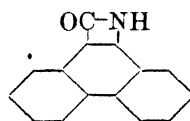
Benrath, Meyer, *J. prakt. Chem.*, 1914, **89**, 258.

Schmidt, Lump, *Ber.*, 1910, **43**, 790.

**Phenanthrane.**

9 : 10-Dihydrophenanthrene, q.v.

**Phenanthranil** (Phenanthranilic acid lactam)



$C_{15}H_9ON$  MW, 219

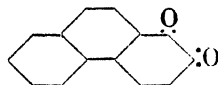
Needles from  $C_6H_6$ . M.p. 241°. Sol.  $C_6H_6$ . Sublimes.  $Na_2CO_3 \rightarrow$  Na salt of acid. Fuming HCl at 170°  $\rightarrow$  9-hydroxyphenanthrene.

N-Acetyl : m.p. 145°.

Et ether :  $C_{17}H_{13}ON$ . MW, 247. Needles from pet. ether. M.p. 110°. Sol. EtOH,  $C_6H_6$ .

Japp, Knox, *J. Chem. Soc.*, 1905, **87**, 692.

**1 : 2-Phenanthraquinone**

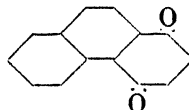


$C_{14}H_8O_2$  MW, 208

Needles from toluene. M.p. 222° decomp. Sol. AcOH. Mod. sol. EtOH,  $C_6H_6$ .  $H_2SO_4 \rightarrow$  blue col. changing rapidly to green.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 1896.

**1 : 4-Phenanthraquinone**

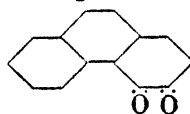


$C_{14}H_8O_2$  MW, 208

Yellow needles from ligroin. M.p. 155°. Sol. EtOH,  $C_6H_6$ , AcOH.  $H_2SO_4 \rightarrow$  violet-red col.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2469.

**3 : 4-Phenanthraquinone**

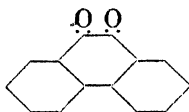


$C_{14}H_8O_2$  MW, 208

Red needles from  $C_6H_6$ -ligroin. M.p.  $133^\circ$  decomp.  $H_2SO_4 \rightarrow$  Prussian-blue col. changing to chrome-green. Red.  $\rightarrow$  morphol.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 946.

### 9 : 10-Phenanthraquinone



$C_{14}H_8O_2$

MW, 208

Orange-yellow needles. M.p.  $206-207.5^\circ$  ( $205^\circ$ ,  $202^\circ$ ). B.p. above  $360^\circ$ . Sol.  $Et_2O$ , hot AcOH. Spar. sol. EtOH,  $C_6H_6$ , AcOEt. Insol.  $H_2O$ . Sol. conc.  $H_2SO_4$  with dull green col. Sublimes in orange-red plates.  $NaHSO_3$  gives unstable bisulphite comp. Ox.  $\rightarrow$  diphenic acid. Red.  $\rightarrow$  phenanthrahydroquinone.

Monoxime : m.p.  $158^\circ$ .

Dioxime : m.p.  $202^\circ$  decomp. (rapid heat).

Me ether : m.p.  $222-3^\circ$ . Di-Me ether : m.p.  $145-6^\circ$ . Diacetyl deriv. : m.p.  $184^\circ$ .

Monosemicarbazone : m.p. about  $220^\circ$  decomp.

Mono-p-nitrophenylhydrazone : m.p.  $245^\circ$ .

Lewis, Gibbs, U.S.P., 1,288,431, (*Chem. Abstracts*, 1919, **13**, 451).

Tseng, Chu, *Chem. Abstracts*, 1934, **28**, 3730.

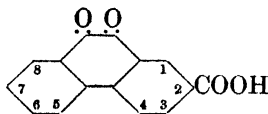
Selden, E.P., 170,022, (*Chem. Abstracts*, 1922, **16**, 1137).

Scholl, Schwarzer, *Ber.*, 1922, **55**, 324.

Auwers, *Ann.*, 1911, **378**, 210.

Kenner, Wilson, *J. Chem. Soc.*, 1927, 1111.

### Phenanthraquinone-2-carboxylic Acid



$C_{15}H_{10}O_4$

MW, 252

Red needles from AcOH. Does not melt below  $310^\circ$ . Spar. sol. hot EtOH, hot  $Me_2CO$ . Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ .

Nitrile :  $C_{15}H_7O_2N$ . MW, 233. Reddish-yellow leaflets from AcOH. M.p.  $290^\circ$ .

Liebermann, Kardos, *Ber.*, 1913, **46**, 201.

Werner, *Ann.*, 1902, **321**, 356.

### Phenanthraquinone-3-carboxylic Acid.

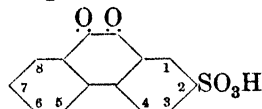
Reddish-yellow cryst. from hot AcOH. Does not melt below  $315^\circ$ .

Amide :  $C_{15}H_9O_3N$ . MW, 251. Orange-red needles. M.p.  $290^\circ$ .

Nitrile :  $C_{15}H_7O_2N$ . MW, 233. Orange cryst. from AcOH. M.p.  $282-3^\circ$ .

Werner, *Ann.*, 1902, **321**, 355.

### Phenanthraquinone-2-sulphonic Acid



$C_{14}H_8O_5S$

MW, 288

Me ester :  $C_{15}H_{10}O_5S$ . MW, 302. Yellow leaflets from AcOH. M.p.  $196-7^\circ$  ( $192-192.5^\circ$ ).

Chloride :  $C_{14}H_7O_4ClS$ . MW, 306.5. Yellow leaflets or needles from AcOH. M.p.  $245-6^\circ$  decomp.

Sandqvist, *Ann.*, 1911, **379**, 89.

### Phenanthraquinone-3-sulphonic Acid.

Me ester : orange-yellow cryst. from AcOH. M.p.  $235^\circ$ .

Chloride : yellowish-red needles. M.p.  $257^\circ$  ( $228-32^\circ$  decomp.).

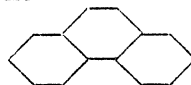
Sandqvist, *Ann.*, 1913, **398**, 136.

Werner, *Ann.*, 1902, **321**, 341.

### Phenanthraquinol.

See Phenanthrahydroquinone.

### Phenanthrene



$C_{14}H_{10}$

MW, 178

Constituent of crude anthracene fraction of coal tar. Plates from EtOH. M.p.  $101^\circ$  ( $98.7-99.5^\circ$ ,  $96.5-97.5^\circ$ ). B.p.  $340^\circ$  ( $332^\circ$ ),  $210-15^\circ/12$  mm. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $Me_2CO$ ,  $CS_2$ ,  $CCl_4$ . Mod. sol. EtOH, MeOH, AcOH, pet. ether. Insol.  $H_2O$ . Sols. show blue fluor.  $D_4^{20}$  0.9800.  $n_D^{20}$  1.59427. Sublimes in leaflets. Ox.  $\rightarrow$  9 : 10-phenanthraquinone  $\rightarrow$  diphenic acid.

Picrate : m.p.  $144^\circ$  ( $132.8^\circ$ ).

$C_{14}H_{10} + C_6H_2Cl(NO_2)_3 \cdot 2 : 1 : 3 : 5 : 82.4^\circ$ .

$C_{14}H_{10}, C_6H_3(NO_2)_3 \cdot 1 : 3 : 5$  : m.p.  $158^\circ$

$C_{14}H_{10}, C_6H_2(NO_2)_3NH_2$  : m.p.  $160-2^\circ$ .

Zelinsky, Titz, Gaverdovshaia, *Ber.*, 1926, **59**, 2590.

Clar, *Ber.*, 1932, **65**, 846, 1411.

Hönig, *Z. Elektrochem.*, 1929, **35**, 847.

Ruzicka, *Helv. Chim. Acta*, 1934, **17**, 473.

Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 557.

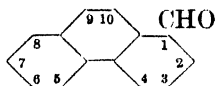
Nikolaev, *Chem. Abstracts*, 1935, **29**, 7972.

Fieser, Herschberg, *J. Am. Chem. Soc.*, 1935, **57**, 1508.

Iljinski, *Chem. Zentr.*, 1932, **I**, 1191.

**Phenanthrene-acetic Acid.**

See Phenanthrylacetic Acid.

**Phenanthrene-1-aldehyde** $C_{15}H_{10}O$ 

MW, 206

M.p. 110–11°.

Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1382.**Phenanthrene-2-aldehyde.**

Needles. M.p. 59–59.5°. Sol. EtOH, MeOH.

Oxime : m.p. 194–5°.

Semicarbazone : m.p. 281–2°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2996.**Phenanthrene-3-aldehyde.**Leaflets from  $C_6H_6$ -pet. ether. M.p. 79.5–80°. Sol. EtOH, MeOH.

Oxime : m.p. 145–145.5°.

Semicarbazone : m.p. 274–5°.

See previous reference.

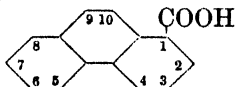
**Phenanthrene-9-aldehyde.**

Yellow prisms from EtOH. M.p. 100–1°. B.p. 231–3°/12 mm. Mod. sol. EtOH.

Oxime : m.p. 157–157.5° (156–7°).

Semicarbazone : m.p. 221–2° (222–222.5°).

p-Nitrophenylhydrazone : m.p. 265°.

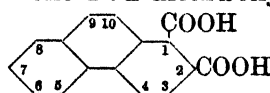
Hinkel, Ayling, Beynon, *J. Chem. Soc.*, 1936, 344.Miller, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 769.**Phenanthrene-1-carboxylic Acid (1-Phenanthroic acid)** $C_{15}H_{10}O_2$ 

MW, 222

Needles from EtOH. M.p. 232–3°. Sol.

 $C_6H_6$ .*Me ester* :  $C_{16}H_{12}O_2$ . MW, 236. M.p. 57°. Sol. ord. org. solvents.*Amide* :  $C_{15}H_{11}ON$ . MW, 221. Plates from AcOH.Aq. M.p. 284°.*Nitrile* :  $C_{15}H_9N$ . MW, 203. Needles from EtOH. M.p. 128°.*Anilide* : m.p. 245°.Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1381.Fieser, *J. Am. Chem. Soc.*, 1932, **54**, 4110.**Phenanthrene-2-carboxylic Acid (2-Phenanthroic acid).**

Needles from AcOH. M.p. 258.5–260°. Sol. EtOH, AcOH.

*Me ester* : needles from MeOH. M.p. 96–96.5°.*Et ester* :  $C_{17}H_{14}O_2$ . MW, 250. Needles from pet. ether. M.p. 73–73.5°.*Chloride* :  $C_{15}H_9OCl$ . MW, 240.5. M.p. 101–101.5°.*Amide* : needles from  $C_6H_6$ . M.p. 242–3°.*Nitrile* : cryst. from EtOH. M.p. 108–109.5°.*Anilide* : m.p. 217–18°.Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2996; 1930, **52**, 3708.Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 558.**Phenanthrene-3-carboxylic Acid (3-Phenanthroic acid,  $\alpha$ -phenanthrene-carboxylic acid).**Needles from AcOH. M.p. 269° (270°). Sol. EtOH, Et<sub>2</sub>O, AcOH. Insol. H<sub>2</sub>O. Sublimes.*Me ester* : needles from MeOH. M.p. 94.5–95° (97°).*Et ester* : needles from pet. ether. M.p. 56–7°.*Chloride* : m.p. 116–17°.*Amide* : cryst. from EtOH. M.p. 233–4° (227–8°).*Nitrile* : needles from EtOH. M.p. 102°.*Anilide* : needles from Me<sub>2</sub>CO. M.p. 216–17°.Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1930, **52**, 3710; 1933, **55**, 2996.Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 558.Kruber, *Ber.*, 1934, **67**, 1005.**Phenanthrene-9-carboxylic Acid (9-Phenanthroic acid,  $\beta$ -phenanthrene-carboxylic acid).**Cryst. from EtOH. M.p. 252°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Insol. H<sub>2</sub>O. Sublimes.*Me ester* : m.p. 116°.*Et ester* : m.p. 61°.*Chloride* : m.p. 102°. B.p. 240°/13 mm.*Amide* : needles from  $C_6H_6$ . M.p. 232–3° (226°).*Nitrile* : needles from EtOH. M.p. 103°.*Anilide* : m.p. 218°.Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, **55**, 2996.Shopper, *J. Chem. Soc.*, 1933, 39.Bachmann, *J. Am. Chem. Soc.*, 1934, **56**, 1366.**Phenanthrene-1 : 2-dicarboxylic Acid** $C_{16}H_{10}O_4$ 

MW, 266

*Anhydride* :  $C_{16}H_8O_3$ . MW, 248. Yellow needles from  $Ac_2O$ . M.p. 311–13°.

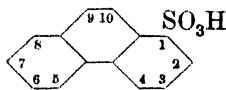
Fieser, Hershberg, *J. Am. Chem. Soc.*, 1935, **57**, 1508, 1853.

### Phenanthrene-1 : 7-dicarboxylic Acid.

*Di-Me ester* :  $C_{18}H_{14}O_4$ . MW, 294. Cryst. from MeOH. M.p. 151–2°.

Ruzicka, Graaff, Hosking, *Helv. Chim. Acta*, 1931, **14**, 238.

### Phenanthrene-1-sulphonic Acid



$C_{14}H_{10}O_3S$

MW, 258

*p-Toluidine salt* : needles. M.p. 267°.

*Me ester* :  $C_{15}H_{12}O_3S$ . MW, 272. Plates from MeOH. M.p. 102°.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.

### Phenanthrene-2-sulphonic Acid.

Cryst. +  $1H_2O$ . M.p. about 150°. Sol. hot toluene, hot  $PhNO_2$ .

*p-Toluidine salt* : m.p. 291°.

*Me ester* : (i) m.p. 101.5°. (ii) Leaflets. M.p. 96–8°. Sol. MeOH, EtOH.

*Et ester* :  $C_{16}H_{14}O_3S$ . MW, 286. Yellowish-brown leaflets from EtOH. M.p. 88.5°.

*Chloride* :  $C_{14}H_9O_2ClS$ . MW, 276.5. Leaflets from AcOH. M.p. 156°.

*Amide* :  $C_{14}H_{11}O_2NS$ . MW, 257. Leaflets from EtOH. M.p. 253–4°.

*Anilide* : m.p. 157–8°.

Sandqvist, *Ann.*, 1911, **379**, 79.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.

Ioffe, *Chem. Abstracts*, 1934, **28**, 1694.

### Phenanthrene-3-sulphonic Acid ( $\alpha$ -Phenanthrenesulphonic acid).

Cryst. from  $H_2O \rightarrow$  two hydrates. (i) +  $1H_2O$ , m.p. 120–1°; (ii) +  $2H_2O$ , m.p. 88–9°. M.p. anhyd. 175–6°.

*p-Toluidine salt* : m.p. 222°.

*Me ester* : cryst. from EtOH. M.p. 119–20°.

*Et ester* : leaflets or needles. M.p. 107–8°. Sol. EtOH,  $C_6H_6$ .

*Chloride* : yellow leaflets from  $C_6H_6$ . M.p. 110–11° (108.5°); solidifies, and remelts at 114°.

*Bromide* :  $C_{14}H_9O_2BrS$ . MW, 321. Yellow plates from ligroin. M.p. 140°.

*Amide* : leaflets from EtOH.Aq. M.p. 189.5–190°.

Sandqvist, *Ann.*, 1909, **369**, 104; 1913, **398**, 136.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.

### Phenanthrene-9-sulphonic Acid ( $\beta$ -Phenanthrenesulphonic acid).

Leaflets or needles +  $2H_2O$  from  $H_2O$  or  $C_6H_6$ . M.p. 134°, 174° anhyd.

*p-Toluidine salt* : needles. M.p. 235°.

*Me ester* : leaflets from MeOH. M.p. 106°.

*Et ester* : cryst. from EtOH. M.p. 108°.

*Chloride* : yellow needles from AcOH. M.p. 127° (125.5°).

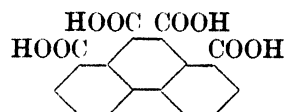
*Amide* : needles from EtOH. M.p. 193.5°.

Sandqvist, *Ann.*, 1912, **392**, 76.

Fieser, *J. Am. Chem. Soc.*, 1929, **51**, 2464.

Ioffe, *Chem. Abstracts*, 1934, **28**, 1694.

### Phenanthrene - 1 : 8 : 9 : 10 - tetracarboxylic Acid



$C_{18}H_{10}O_8$

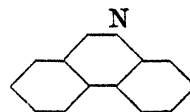
MW, 354

*Tetra-Me ester* :  $C_{22}H_{18}O_8$ . MW, 410. Yellow cryst. M.p. 181–3°.

*Di-anhydride* :  $C_{18}H_6O_6$ . MW, 318. Brownish-yellow needles. Does not melt below 400°.

Zinke, Hauswirth, Grimm, *Monatsh.*, 1931, **57**, 405.

### Phenanthridine (3 : 4 - Benzquinoline, 2 : 3 : 4 : 5-dibenzpyridine)



$C_{13}H_9N$

MW, 179

Needles from EtOH.Aq. M.p. 106°. B.p. 349°/769 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ ,  $CS_2$ . Spar. sol.  $H_2O$  with blue fluor. Spar. volatile in steam.

$B, HCl, HgCl_2$  : m.p. 197°.

$B, H_2PtCl_6$  : m.p. above 225°.

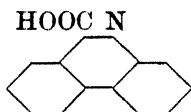
*Methiodide* : m.p. 204.5°.

*Picrate* : m.p. 220°.

Sielisch, Sandke, *Ber.*, 1933, **66**, 433.

Pyl, *Ber.*, 1927, **60**, 287.

## Phenanthridine-9-carboxylic Acid

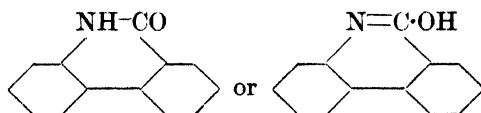
 $C_{14}H_9O_2N$ 

MW, 223

Micro-cryst. Sol. EtOH,  $Me_2CO$ . Spar. sol.  $C_6H_6$ . Insol.  $H_2O$ , pet. ether. Heat  $\rightarrow$  phenanthridine.

*Et ester*:  $C_{16}H_{13}O_2N$ . MW, 251. Needles from pet. ether. M.p. 57–8°.

Walls, *J. Chem. Soc.*, 1934, 108.

Phenanthridone (*Phenanthridol*, 10-hydroxy-phenanthridine) $C_{13}H_9ON$ 

MW, 195

Needles from EtOH. M.p. 293°. Spar. sol. EtOH,  $Et_2O$ , AcOH. Zn dist.  $\rightarrow$  phenanthridine.

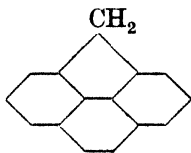
Oyster, Adkins, *J. Am. Chem. Soc.*, 1921, 43, 210.

Meyer, Hofmann, *Monatsh.*, 1916, 37, 701.

Walls, *J. Chem. Soc.*, 1934, 108; 1935, 1407.

Heidenreich, Tust, U.S.P., 1,880,441, (*Chem. Abstracts*, 1933, 27, 516).

See also Sielich, Sandke, *Ber.*, 1933, 66, 434.

Phenanthrindene (*Phenanthrylenemethane*) $C_{15}H_{10}$ 

MW, 190

Needles from EtOH. M.p. 116°. B.p. 353°.

*Benzylidene deriv.*: m.p. 108°.

*Picrate*: m.p. 166°.

Kruber, *Ber.*, 1934, 67, 1004.

## Phenanthroic Acid.

See Phenanthrene-carboxylic Acid.

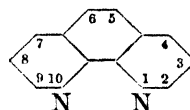
## Phenanthrol.

See Hydroxyphenanthrene.

## Phenanthrol-carboxylic Acid.

See Hydroxyphenanthrene-carboxylic Acid.

## o-Phenanthroline (1 : 10-Phenanthroline)

 $C_{12}H_8N_2$ 

MW, 180

Cryst. +  $1H_2O$  from  $H_2O$ , m.p. 91.5°; cryst. from  $C_6H_6$ , m.p. 98–100°. B.p. above 300°. Sol. EtOH,  $C_6H_6$ ,  $Me_2CO$ . Insol. pet. ether. Ferrous salts  $\rightarrow$  red col. Forms metallic derivs.

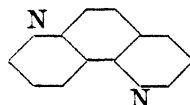
Tartarini, Samaja, *Chem. Abstracts*, 1933, 27, 5741.

Smith, Getz, *Chemical Reviews*, 1935, 16, 113.

Willink, Wibaut, *Rec. trav. chim.*, 1935, 54, 282.

Smith, *J. Am. Chem. Soc.*, 1930, 52, 402.

## m-Phenanthroline (1 : 7-Phenanthroline)

 $C_{12}H_8N_2$ 

MW, 180

Needles. M.p. 65.5°, anhyd. 78–78.5°. B.p. above 360°. Sol. EtOH, hot  $H_2O$ . Insol.  $Et_2O$ ,  $C_6H_6$ , ligroin.

Knüppel, *Ber.*, 1896, 29, 707.

See also Smith, *J. Am. Chem. Soc.*, 1930, 52, 397.

p-Phenanthroline ( $\psi$ -Phenanthroline, 4 : 7-phenanthroline) $C_{12}H_8N_2$ 

MW, 180

Needles from  $H_2O$ . M.p. anhyd. 173°. Sol. EtOH,  $CHCl_3$ . Mod. sol. hot  $H_2O$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ .

*Hydrochloride*: does not melt below 315°.

*Sulphate*: m.p. 233–4°.

*Chromate*: decomp. at 225–30°.

*Picrate*: m.p. 255–6°.

*Platinochloride*: does not melt below 310°.

*Methiodide*: m.p. 268–9°.

*Di-methiodide*: m.p. 271°.

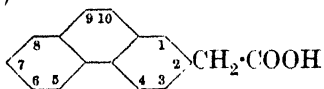
Matsumura, *J. Am. Chem. Soc.*, 1930, 52, 3196.

Smith, *ibid.*, 402.

## Phenanthrone.

Keto form of 9-Hydroxyphenanthrene, *q.v.*



**2-Phenanthrylacetic Acid** (*Phenanthrene-2-acetic acid*)

$C_{16}H_{12}O_2$  MW, 236

Needles. M.p. 183.5–184.5°.

*Me ester*:  $C_{17}H_{14}O_2$ . MW, 250. Needles. M.p. 78–78.5°.

*Nitrile*:  $C_{16}H_{11}N$ . MW, 217. M.p. 106–106.5°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, 55, 2998.

**3-Phenanthrylacetic Acid** (*Phenanthrene-3-acetic acid*).

Leaflets. M.p. 177–177.5°.

*Me ester*: liq. *Picrate*: m.p. 103.5–104°.

*Nitrile*: m.p. 84.5–85°.

See previous reference.

**9-Phenanthrylacetic Acid** (*Phenanthrene-9-acetic acid*).

Leaflets. M.p. 220–1° (213–15°).

*Me ester*: m.p. 75–75.5°.

*Amide*:  $C_{16}H_{13}ON$ . MW, 235. Leaflets. M.p. 250–2°.

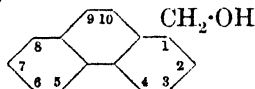
*Nitrile*: m.p. 96.5–97°.

See previous reference and also

Willgerodt, Albert, *J. prakt. Chem.*, 1911, 84, 387.

**Phenanthrylamine.**

See Aminophenanthrene.

**1-Phenanthrylcarbinol** (*1-Hydroxymethylphenanthrene*)

$C_{15}H_{12}O$  MW, 208

M.p. 165°.

Bachmann, *J. Am. Chem. Soc.*, 1935, 57, 1382.

**2-Phenanthrylcarbinol** (*2-Hydroxymethylphenanthrene*).

Needles from  $C_6H_6$ -pet. ether. M.p. 125–125.5°.

Mosettig, van de Kamp, *J. Am. Chem. Soc.*, 1933, 55, 2998.

**3-Phenanthrylcarbinol** (*3-Hydroxymethylphenanthrene*).

Prisms from  $C_6H_6$ -pet. ether. M.p. 103–103.5°.

See previous reference.

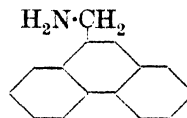
**9-Phenanthrylcarbinol** (*9-Hydroxymethylphenanthrene*).

Needles from  $C_6H_6$ -pet. ether. M.p. 149–149.5°.

Bachmann, *J. Am. Chem. Soc.*, 1934, 56, 1366.

**Phenanthrylene-methane.**

See Phenanthrindene.

**9-Phenanthrylmethylamine** (*9-Aminomethylphenanthrene*)

$C_{15}H_{13}N$  MW, 207

Cryst. from  $Et_2O$ -ligroin. M.p. 107°.

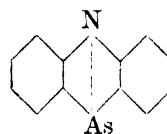
*N-Acetyl*: m.p. 182–5°.

*N-Benzoyl*: m.p. 167°.

*N-Benzylidene*: m.p. 103.5°.

*Picrate*: m.p. 241° decomp.

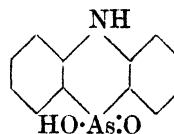
Shoppee, *J. Chem. Soc.*, 1933, 40.

**Phenarsazine** (*Phenazarsine*)

$C_{12}H_8NaAs$  MW, 241

M.p. about 310°. Sol.  $PhNO_2$ . Spar. sol. hot xylene.

Wieland, Rheinheimer, *Ann.*, 1921, 423, 16.

**Phenarsazinic Acid** (*Phenazarsinic acid*)

$C_{12}H_{10}O_2NaAs$  MW, 275

Cryst. from  $H_2O$ . M.p. 365–6° decomp.

*B,HCl*: m.p. 208°.

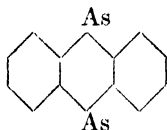
Kappelmeier, *Rec. trav. chim.*, 1930, 49, 83 (*Bibl.*).

Sergeev, Gorkii, *Chem. Abstracts*, 1932, 26, 2195.

Wieland, Rheinheimer, *Ann.*, 1921, 423, 22.

See also Razuvaev, Koton, *Chem. Abstracts*, 1933, 27, 984.

**Phenarsine** (*Arsanthrene*, *diarseno-9:10-anthracene*)



$C_{12}H_8As_2$  MW, 302

Orange-yellow leaflets. M.p. about  $340^\circ$ . Spar. sol. AcOH, Py.

Kalb, *Ann.*, 1921, **423**, 66.

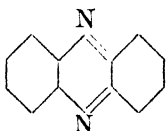
**Phenazarsine.**

See Phenarsazine.

**Phenazarsinic Acid.**

See Phenarsazinic Acid.

**Phenazine** (*Azophenylene*)



$C_{12}H_8N_2$  MW, 180

Yellowish-red needles, from AcOH. M.p.  $171^\circ$  ( $170^\circ$ ). B.p. above  $360^\circ$ . Sol. hot EtOH. Mod. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Sublimes. Volatile in steam.

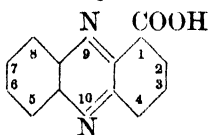
Eckert, Steiner, *Monatsh.*, 1914, **35**, 1153.  
Zerewitinoff, Ostromisslensky, *Ber.*, 1911, **44**, 2402.

Kehrmann, Mermod, *Helv. Chim. Acta*, 1927, **10**, 62.

Beschke, *Ann.*, 1913, **398**, 298.

Clemo, McIlwain, *J. Chem. Soc.*, 1934, 1993.

**Phenazine-1-carboxylic Acid**



$C_{13}H_8O_2N_2$  MW, 224

Yellow needles from EtOH. M.p.  $239^\circ$ .

Amide:  $C_{13}H_9ON_3$ . MW, 223. M.p.  $234^\circ$ .

See last reference above and also

Kögl, Tönnis, Groenewegen, *Ann.*, 1932, **497**, 277.

**Phenazine-2-carboxylic Acid.**

Yellow needles from Me<sub>2</sub>CO. M.p.  $292-3^\circ$ .

Amide:  $C_{13}H_9ON_3$ . MW, 223. M.p.  $312^\circ$ .

Clemo, McIlwain, *J. Chem. Soc.*, 1935, 741.

Kögl, Tönnis, Groenewegen, *Ann.*, 1932, **497**, 288.

Dict. of Org. Comp.—III.

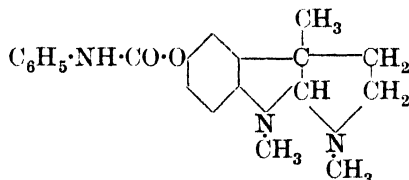
**Phenazone.**

See Antipyrine.

**Phenazoxine.**

See Phenoxazine.

**Pheneserine** (*Eseroline phenylurethane*)



$C_{20}H_{23}O_2N_3$  MW, 337

Prisms. M.p.  $150^\circ$ . NaOH  $\longrightarrow$  eseroline.

Methiodide: m.p.  $198^\circ$ .  $[\alpha]_D -92.8^\circ$  in EtOH.

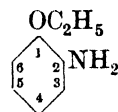
Picrate: non-cryst. M.p. about  $100^\circ$ .

Polonovski, *Bull. soc. chim.*, 1916, **19**, 46.

**Phenetetrol.**

See Apionol.

**o-Phenetidine** (*o-Ethoxyaniline*, *o-amino-phenetole*, *o-aminophenol ethyl ether*)



$C_8H_{11}ON$  MW, 137

B.p.  $228^\circ$  ( $224-9^\circ$ ).  $k = 4.64 \times 10^{-10}$  at  $20^\circ$ .

$B_2C_{10}H_7SO_3H$  (1): m.p.  $185^\circ$ .

$B_2C_{10}H_7SO_3H$  (2): m.p.  $197^\circ$ .

N-Formyl: form-o-phenetidide. M.p.  $62^\circ$ . B.p.  $292^\circ$ .

N-Acetyl: acet-o-phenetidide, o-ethoxyacetanilide.  $C_{10}H_{13}O_2N$ . MW, 179. M.p.  $79^\circ$ .

N-Lauryl: m.p.  $69.7-70^\circ$ .

N-Myristyl: m.p.  $77^\circ$ .

N-Palmityl: m.p.  $82-3^\circ$ .

N-Stearyl: m.p.  $84.7^\circ$ .

N-Benzoyl: benz-o-phenetidide. M.p.  $104.2^\circ$ .

N-1-Naphthalenesulphonyl: m.p.  $185^\circ$ .

N-2-Naphthalenesulphonyl: m.p.  $197^\circ$ .

Zavelskii, Fomenko, Krolik, *Chem. Abstracts*, 1935, **29**, 467.

Li, Adams, *J. Am. Chem. Soc.*, 1935, **57**, 1568.

Birosel, Huang, *Chem. Abstracts*, 1933, **27**, 5727.

**m-Phenetidine** (*m-Ethoxyaniline*, *m-amino-phenetole*, *m-aminophenol ethyl ether*).

B.p.  $248^\circ$ ,  $127-8^\circ/11$  mm.

N-Formyl: form-m-phenetidide. Greyish-blue cryst. M.p.  $52^\circ$ .

N-Acetyl: acet-*m*-phenetidide. Greyish plates. M.p. 96–7°.

N-Benzoyl: see under *m*-Hydroxybenzanilide.

N-Toluene-*p*-sulphonyl: m.p. 157°.

Picrate: m.p. 158°.

Reverdin, Lokietek, *Bull. soc. chim.*, 1915, 17, 407.

**p-Phenetidine** (*p*-Ethoxyaniline, *p*-aminophenetole, *p*-aminophenol ethyl ether).

M.p. 2–4°. B.p. 254·2–254·7°.  $k = 2 \cdot 15 \times 10^{-9}$  at 15°.  $D_4^{15} 1 \cdot 0652$ . Spar. volatile in steam.

*B*<sub>2</sub>HCl: m.p. 234°. Sol. H<sub>2</sub>O. Sublimes.

*B*<sub>2</sub>(COOH)<sub>2</sub>: m.p. 201°.

*B*<sub>2</sub>(COOH)<sub>2</sub>: m.p. 201°.

Tartrate: vinopyrin. M.p. 192°.

N-Formyl: form-*p*-phenetidide. M.p. 68–70°.

N-Acetyl: acet-*p*-phenetidide. Phenacetin. C<sub>10</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 179. M.p. 137–8°. Sol. 1400 parts H<sub>2</sub>O at 15°, 80 parts at boil. Sol. 6 parts Py at 20°, 67 parts Et<sub>2</sub>O at 25°, 20 parts CHCl<sub>3</sub> at 25°, 10 parts Me<sub>2</sub>CO at 30°, 170 parts C<sub>6</sub>H<sub>6</sub> at 30°, 6·5 parts EtOH. Heat of comb. C<sub>p</sub> 1303 Cal. NaOBr + NH<sub>3</sub> → brown col. Forms cryst. K deriv. Antipyretic and antineuralgic. *B*<sub>2</sub>HI: needles. M.p. 147–8°.

N-Lactyl: see under Lactic acid.

N-Glycyl: phenocoll. Needles + 1H<sub>2</sub>O. M.p. 95°, 100·5° anhyd.

N-Lauryl: m.p. 109–10°.

N-Palmityl: m.p. 117·5°.

N-Stearyl: m.p. 112·5°.

Glucoside: m.p. 114°.

N-Furfurylidene: m.p. 72–3°.

N-Benzoyl: see under *p*-Hydroxybenzanilide.

N-Mandelyl: see Amygdophenine.

N-1-Naphthalenesulphonyl: m.p. 201–2°.

N-2-Naphthalenesulphonyl: m.p. 207°.

N-Me: see N-Methyl-*p*-phenetidine.

Täufel, Wagner, Dünwald, *Z. Elektrochem.*, 1928, 34, 115.

Grether, U.S.P., 1,722,417, (*Chem. Abstracts*, 1929, 23, 4483).

Birosel, Huang, *Chem. Abstracts*, 1933, 27, 5727.

Ehrhardt, *Ber.*, 1897, 30, 2015.

Byk, D.R.P., 264,263, (*Chem. Zentr.*, 1913, II, 1179).

Zernik, *Chem. Zentr.*, 1908, I, 1203.

Aoyama, Eguchi, Tashiro, *Chem. Abstracts*, 1935, 29, 5427.

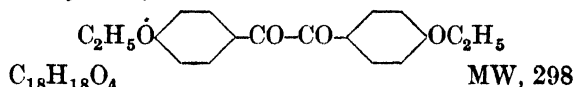
Zavelskii, Fomenko, Krolik, *ibid.*, 467.

West, *J. Chem. Soc.*, 1925, 494.

Hinsberg, *Ann.*, 1899, 305, 278.

Tauber, D.R.P., 85,988.

**p-Phenetil** (*Di*-[4-ethoxy]-benzoyl, 4:4'-di-ethoxybenzil)



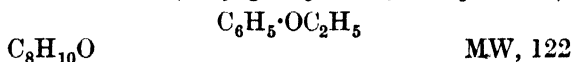
Prisms from EtOH. M.p. 149°.

Monoxime: m.p. 136°.

Di-phenylhydrazone: m.p. 171°.

Vorländer, *Ber.*, 1911, 44, 2464.

**Phenetole** (*Ethyl phenyl ether*, *ethoxybenzene*)



F.p. –28·6° (–30·2°). M.p. –33°. B.p. 172°, 166°/667·5 mm., 160°/565·5 mm., 92·5°/61·42 mm., 77·5°/31·14 mm., 60°/9·12 mm. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Heat of comb. C<sub>p</sub> 1057·23 Cal.  $D_4^{20} 0 \cdot 9666$ .  $n_D^{18} 1 \cdot 5084$ .

*C*<sub>8</sub>H<sub>10</sub>O, *SbCl*<sub>3</sub>: m.p. 42·2°.

*C*<sub>8</sub>H<sub>10</sub>O, *SbBr*<sub>3</sub>: m.p. 48·8°.

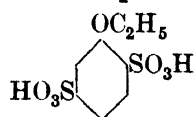
Rodionov, *Bull. soc. chim.*, 1929, 45, 109.

Zerbe, D.R.P., 563,969, (*Chem. Abstracts*, 1933, 27, 4812).

Sowa, Hennion, Nieuwland, *J. Am. Chem. Soc.*, 1935, 57, 710.

Finzi, *Chem. Abstracts*, 1925, 19, 2648.

**Phenetole-2:5-disulphonic Acid**



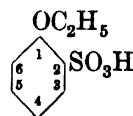
$C_8H_{10}O_7S_2$  MW, 282

Dichloride: C<sub>8</sub>H<sub>8</sub>O<sub>6</sub>Cl<sub>2</sub>S<sub>2</sub>. MW, 319. Plates from Et<sub>2</sub>O. M.p. 106–8°.

Diamide: C<sub>8</sub>H<sub>12</sub>O<sub>5</sub>N<sub>2</sub>S<sub>2</sub>. MW, 280. Needles from H<sub>2</sub>O. M.p. 233°.

Zander, *Ann.*, 1879, 198, 25.

**Phenetole-o-sulphonic Acid**



$C_8H_{10}O_4S$  MW, 202

Cryst. mass.

Chloride: C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>ClS. MW, 220·5. Plates from pet. ether. M.p. 65–6°.

Amide: C<sub>8</sub>H<sub>11</sub>O<sub>3</sub>NS. MW, 201. Needles from H<sub>2</sub>O. M.p. 163°.

Anilide: m.p. 158°.

Phenylhydrazide: m.p. 132–3°.

Moody, *Chem. News*, 1893, 67, 35.

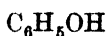
Gattermann, *Ber.*, 1899, 32, 1154.

**Phenetole-*m*-sulphonic Acid.**Cryst. Sol.  $\text{H}_2\text{O}$ , EtOH.*Et ester* :  $\text{C}_{10}\text{H}_{14}\text{O}_4\text{S}$ . MW, 230. Oil. Decomp. on dist. Volatile in steam.*Chloride* : needles from  $\text{Et}_2\text{O}$ . M.p.  $38^\circ$ . Sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Spar. sol. EtOH.*Amide* : needles from  $\text{H}_2\text{O}$ . M.p.  $131^\circ$  ( $126^\circ$ ).Lagai, *Ber.*, 1892, 25, 1836.**Phenetole-*p*-sulphonic Acid.***Chloride* : prisms or leaflets from  $\text{Et}_2\text{O}$ . M.p.  $39^\circ$  ( $36.5^\circ$ ). Sol. EtOH,  $\text{Et}_2\text{O}$ .*Amide* : needles from  $\text{H}_2\text{O}$ , plates from EtOH. M.p.  $150^\circ$  ( $149^\circ$ ).Lagai, *Ber.*, 1892, 25, 1837.Moody, *Chem. News*, 1892, 65, 247.Gattermann, *Ber.*, 1899, 32, 1155.***p*-Phenetylurea.**

See Dulcin.

**Phenobarbital.**

See Luminal.

**Phenocoll.**See under *p*-Phenetidine.**Phenol (Hydroxybenzene, carbolic acid)**

MW, 94

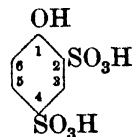
Needles. F.p.  $41^\circ$  ( $40.5^\circ$ ). M.p.  $43^\circ$ . B.p.  $182^\circ$ ,  $159.7^\circ/400\text{ mm.}$ ,  $90.2^\circ/25\text{ mm.}$  Sol. EtOH,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$ .  $D_4^{25}$  1.0710.  $n_D^{25}$  1.5509.  $k = 1.3 \times 10^{-10}$  at  $25^\circ$ .  $\text{FeCl}_3 \rightarrow$  violet col. Turns pink in air. Corrosive poison. Heat of comb.  $\text{C}_p$  732.5 Cal.,  $\text{C}_v$  734.2 ( $731-9$ ) Cal. Volatile in steam.

 $\text{C}_6\text{H}_5\text{O}, \frac{1}{2}\text{H}_2\text{O}$  : m.p.  $16^\circ$ . $\text{C}_6\text{H}_5\text{O}, 2\text{SbCl}_3$  : m.p.  $37^\circ$ . $\text{C}_6\text{H}_5\text{O}, 2\text{SbBr}_3$  : m.p.  $66.5^\circ$ . $2\text{C}_6\text{H}_5\text{O}, \text{CH}_3\cdot\text{CO}\cdot\text{CH}_3$  : m.p.  $15^\circ$ . $2\text{C}_6\text{H}_5\text{O}, \text{CH}_3\cdot\text{CO}\cdot\text{NH}_2$  : m.p.  $40.8^\circ$ . $\text{C}_6\text{H}_5\text{O}, \text{CCl}_3\cdot\text{COOH}$  : m.p.  $37.6^\circ$ . $2\text{C}_6\text{H}_5\text{O}, \text{CO}(\text{NH}_2)_2$  : m.p.  $61^\circ$ . $\text{C}_6\text{H}_5\text{O}, 2\text{CH}_3\cdot\text{NH}_2$  : m.p.  $8.5-9.5^\circ$ .*Picrate* : m.p.  $83.1^\circ$ .

*Phenylurethane* : needles from  $\text{C}_6\text{H}_6$ . M.p.  $126^\circ$ . Dist.  $\rightarrow$  phenyl isocyanate.  $\text{NH}_3 \rightarrow$  phenylurea.

*1-Naphthylurethane* : m.p.  $136-7^\circ$ .*Sulphite* :  $\text{C}_{12}\text{H}_{10}\text{O}_3\text{S}$ . MW, 234. B.p.  $185^\circ/15\text{ mm.}$ ,  $143^\circ/0.7\text{ mm.}$ *Hydrogen sulphate* : see Phenyl hydrogen sulphate.*Hydrogen phosphite* :  $\text{C}_{12}\text{H}_{11}\text{O}_3\text{P}$ . MW, 234. B.p.  $218-19^\circ/25\text{ mm.}$ *Phosphite* : triphenyl phosphite.  $\text{C}_{18}\text{H}_{15}\text{O}_3\text{P}$ . MW, 310. B.p.  $360^\circ$ ,  $235^\circ/18\text{ mm.}$ *Dihydrogen phosphate* :  $\text{C}_6\text{H}_7\text{O}_4\text{P}$ . MW, 174. M.p.  $99.5^\circ$  ( $97-8^\circ$ ).*Hydrogen phosphate* : diphenyl phosphate.  $\text{C}_{12}\text{H}_{11}\text{O}_4\text{P}$ . MW, 250. Needles or leaflets +  $2\text{H}_2\text{O}$ . M.p.  $51^\circ$ , anhyd.  $70^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , hot  $\text{H}_2\text{O}$ .*Phosphate* : see Triphenyl phosphate.*Orthosilicate* :  $\text{C}_{24}\text{H}_{20}\text{O}_4\text{Si}$ . MW, 400. M.p.  $47-8^\circ$ . B.p.  $417-20^\circ$ .*Borate* : triphenyl borate.  $\text{C}_{18}\text{H}_{15}\text{O}_3\text{B}$ . MW, 290. Needles from pet. ether. M.p.  $50^\circ$ .*Carbonate* : see Diphenyl carbonate.*d- $\alpha$ -Glucoside* :  $\text{C}_{12}\text{H}_{16}\text{O}_6$ . MW, 256. M.p.  $174-5^\circ$ .  $[\alpha]_D^{20} - 71^\circ$  in  $\text{H}_2\text{O}$ .*d- $\beta$ -Glucoside* : m.p.  $139-41^\circ$ .  $[\alpha]_D^{20} - 39.85^\circ$  in  $\text{H}_2\text{O}$ .Cappelli, *Gazz. chim. ital.*, 1918, 48, ii, 107.Vorozhtzov, Oshuev, *Chem. Abstracts*, 1934, 28, 1027 (Review).I.G., D.R.P., 613,726, (*Chem. Abstracts*, 1935, 29, 8298).du Pont, U.S.P., 2,007,327, *ibid.*, 5864.Rütgerswerke A.-G., E.P., 427,145, *ibid.*, 6252.Eikhman, Shemyakin, Vozhdaeva, *ibid.*, 2520.Tishchenko, Churbakow, *ibid.*, 2520.Noller, Dutton, *J. Am. Chem. Soc.*, 1933, 55, 424.Mizoshita, *Chem. Abstracts*, 1931, 25, 1812.Dyson, *Chem. Age*, 1926, 14, 70 (Review).Battegay, Denivelle, *Compt. rend.*, 1931, 192, 492.Bernton, *Ber.*, 1922, 55, 3361.**Phenoldiazonium chloride.**

See Hydroxybenzenediazonium chloride.

**Phenol-2 : 4-disulphonic Acid**

MW, 254

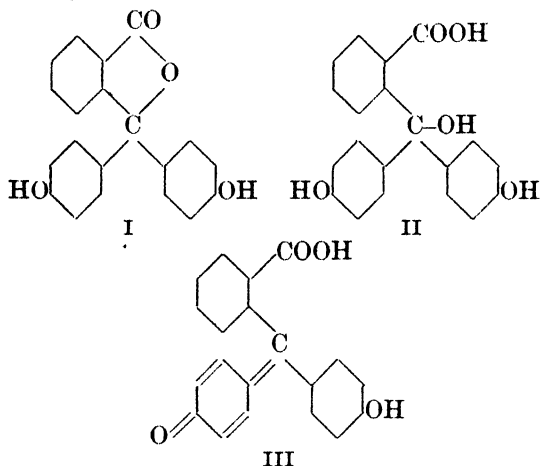
Needles. Decomp. above  $100^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ .*Dichloride* :  $\text{C}_6\text{H}_4\text{O}_5\text{Cl}_2\text{S}_2$ . MW, 291. Plates from  $\text{Et}_2\text{O}$ . M.p.  $89^\circ$  ( $86^\circ$ ).*Diamide* :  $\text{C}_6\text{H}_8\text{O}_5\text{N}_2\text{S}_2$ . MW, 252. Leaflets. M.p.  $239^\circ$ .*Dianilide* : m.p.  $205^\circ$ .Gebauer-Fulnegg, Riesz, *Austrian P.*, 119,960 (*Chem. Abstracts*, 1931, 25, 1262).

**Phenol-2 : 5-disulphonic Acid.**

*Et ether* : phenetole-2 : 5-disulphonic acid.  
*Dichloride* :  $C_8H_8O_5Cl_2S_2$ . MW, 319. Plates from  $Et_2O$ . M.p. 106–8°. *Diamide* :  $C_8H_{12}O_5N_2S_2$ . MW, 280. Needles from  $H_2O$ . M.p. 233°.

Senhofer, *Sitzb. Akad. Wiss. Wien*, 1878, 78, ii, 678.

**Phenolphthalein** (3 : 3-Di-[*p*-hydroxyphenyl]-phthalide,  $\alpha$ -4 : 4'-trihydroxytriphenylmethane-2-carboxylic acid lactone)



I. $C_{20}H_{14}O_4$	MW, 318
II. $C_{20}H_{16}O_5$	MW, 336
III. $C_{20}H_{14}O_4$	MW, 318

**I.**  
 Cryst. M.p. 254° (250–3°). Mod. sol. hot  $H_2O$ . Sol. hot  $Et_2O$ .  $NaOH \rightarrow$  red col. Used as indicator. Red.  $\rightarrow$  phenolphthalin.

*Me ether* :  $C_{21}H_{16}O_4$ . MW, 332. Needles from  $C_6H_6$ . M.p. 198–9°  $\rightarrow$  glassy mass, m.p. 80°.

*Di-Me ether* :  $C_{22}H_{18}O_4$ . MW, 346. Leaflets from  $EtOH$ . M.p. 101–2° (97–9°). Sol. hot  $EtOH$ .

*Di-Et ether* :  $C_{24}H_{22}O_4$ . MW, 374. Leaflets from  $EtOH$ . M.p. 122° (118–20°). Sol.  $C_6H_6$ .

*Diphenyl ether* :  $C_{32}H_{22}O_4$ . MW, 470. Grey cryst. M.p. 105–6°.

*Dibenzyl ether* :  $C_{24}H_{26}O_4$ . MW, 498. Leaflets. M.p. 150°. Sol.  $C_6H_6$ .

*Diacetyl* : m.p. 143°.

*Di-isovaleryl* : m.p. 110°.

*Dibenzoyl* : m.p. 169°.

*Dibenzenesulphonyl* : m.p. 112–13°.

**III.**

*Me ester* :  $C_{21}H_{16}O_4$ . MW, 332. Orange-red needles. M.p. 127–30°. Sol.  $EtOH$ ,  $CHCl_3$ .

*Et ester* : *Et ether*,  $C_{24}H_{22}O_4$ . MW, 374. Yellow cryst. M.p. 98–104°.

Underwood, Barker, *J. Am. Chem. Soc.*, 1930, 52, 4084.

Molle, *Chem. Abstracts*, 1927, 21, 2470 (*Bibl.*).

Day, *J. Am. Chem. Soc.*, 1930, 52, 646.

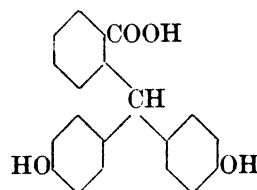
Lund, *J. Chem. Soc.*, 1930, 1844.

Blicke, Swisher, *J. Am. Chem. Soc.*, 1934, 56, 902.

Kavalco, U.S.Ps., 1,940,146, 1,940,494, (*Chem. Abstracts*, 1934, 28, 1366).

Zelinskii, Maksorov, *Ind. Eng. Chem.*, 1931, 24, 63.

**Phenolphthalin** (4 : 4'-Dihydroxytriphenylmethane-2-carboxylic acid)



$C_{20}H_{16}O_4$  MW, 320

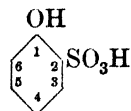
Needles from  $H_2O$ . M.p. 225°. Spar. sol.  $H_2O$ . Ox.  $\rightarrow$  phenolphthalein.

*Et ester* :  $C_{22}H_{20}O_4$ . MW, 348. Leaflets or needles from  $EtOH$ . Aq. M.p. 150–2° (156–8°).

*Di-Me ether* :  $C_{22}H_{20}O_4$ . MW, 348. Needles from  $EtOH$ . M.p. 149–50°.

*Diacetyl* : m.p. 146°.

Baeyer, *Ann.*, 1880, 202, 80.

**Phenol-*o*-sulphonic Acid**

$C_6H_6O_4S$  MW, 174

Cryst. +  $\frac{3}{2}H_2O$ , partly melts at 50° decomp. : +  $H_2O$ , m.p. 145°. Sol.  $H_2O$ .

*K salt*,  $2H_2O$  : m.p. 235–40°.

*Chloride* : *acetyl deriv.*, cryst. from ligroin. M.p. 72.2–72.9°. Sol.  $EtOH$ ,  $C_6H_6$ .

*Anilide* : m.p. 126.5–127.5°. *Acetyl*, m.p. 106–7°.

*p-Toluidide* : m.p. 124–5°. *Acetyl*, m.p. 116–17°.

*Hexamethylenetetramine salt* : m.p. 140°. B.p. 180°.

*Di-hexamethylenetetramine salt* : m.p. 160°.

Tomcsik, *Chem. Abstracts*, 1930, 24, 4517.  
 Anschütz, *Ann.*, 1918, 415, 68.

**Phenol-*m*-sulphonic Acid.**Needles + 2H<sub>2</sub>O. FeCl<sub>3</sub> → violet col.*K* salt, H<sub>2</sub>O: m.p. 200–10°.Chloride: C<sub>6</sub>H<sub>5</sub>O<sub>3</sub>ClS. MW, 192.5. Brown oil. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.Obermiller, *Ann.*, 1911, **381**, 115.Szathmary, *Ber.*, 1910, **43**, 2485.**Phenol-*p*-sulphonic Acid.***Et* ester: benzoyl deriv., m.p. 62°.Fluoride: C<sub>6</sub>H<sub>5</sub>O<sub>3</sub>FS. MW, 176. M.p. 77°.Chloride: C<sub>6</sub>H<sub>5</sub>O<sub>3</sub>ClS. MW, 192.5. Acetyl, m.p. 78°. B.p. 148°/12. Carboxyl, m.p. 75°.

Benzoyl, m.p. 115–16°.

Amide: C<sub>6</sub>H<sub>7</sub>O<sub>3</sub>NS. MW, 173. Cryst. from EtOH. M.p. 176–7°.

Anilide: m.p. 141°. Acetyl, m.p. 126–7°.

Aniline salt: m.p. 112–13°.

*p*-Toluidide: m.p. 151–2°. Acetyl, m.p. 98–9°.

Piperidide: m.p. 132–3°. Acetyl, m.p. 115–16°.

Hexamethylenetetramine salt: m.p. 180–2°.

Di-hexamethylenetetramine salt: m.p. about 190°.

Major, E.P., 328,220, (*Chem. Abstracts*, 1930, **24**, 5509).Steinkopf, D.R.P., 497,242, (*Chem. Abstracts*, 1930, **24**, 3517).Fichter, Tamm, *Ber.*, 1910, **43**, 3036.Anschtz, Molineus, *Ann.*, 1918, **415**, 56.**Phenol-tricarboxylic Acid.**

See 5-Hydroxytrimellitic Acid and Hydroxy-trimesic Acid.

**Phenomydrol.**

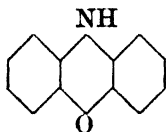
See Phenacylamine.

**Phenoprene.**

See 2-Phenylbutadiene-1:3.

**Phenothioxine.**

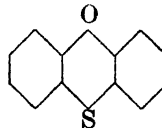
See Phenoxthine.

**Phenoxazine (Phenazoxine)**C<sub>12</sub>H<sub>9</sub>ON

MW, 183

Leaflets from EtOH. M.p. 156° (153–4°). FeCl<sub>3</sub> → bluish-green col. H<sub>2</sub>SO<sub>4</sub> → violet-red col.

Acetyl deriv.: prisms. M.p. 142°.

Cullinane, Davey, Padfield, *J. Chem. Soc.*, 1934, 718.Kehrmann, Neil, *Ber.*, 1903, **47**, 3107.Fresenius, *Z. anal. Chem.*, 1934, **96**, 433 (Review).**Phenoxthine (Phenothioxine, dibenzthioxine)**C<sub>12</sub>H<sub>8</sub>OS

MW, 200

Cryst. from MeOH. M.p. 59° (57.5–58°). B.p. 185–7°/23 mm. H<sub>2</sub>SO<sub>4</sub> → intense violet col.Suter, McKenzie, Maxwell, *J. Am. Chem. Soc.*, 1936, **58**, 717.A.G.F.A., D.R.P., 234,743, (*Chem. Abstracts*, 1911, **5**, 2912).Drew, *J. Chem. Soc.*, 1928, 519.Ferrario, *Bull. soc. chim.*, 1911, **9**, 536.**Phenoxyacetaldehyde (ω-Aldehydoanisole, glycollic aldehyde phenyl ether)**C<sub>6</sub>H<sub>5</sub>O·CH<sub>2</sub>·CHOC<sub>8</sub>H<sub>8</sub>O<sub>2</sub>

MW, 136

Cryst. + 1H<sub>2</sub>O. M.p. 38°. B.p. 215° decomp., 118–19°/30 mm., 101–3°/15 mm. Unstable. D<sub>4</sub><sup>20</sup> 1.1310. n<sub>D</sub><sup>20</sup> 1.5380.

Oxime: m.p. 95°.

Semicarbazone: m.p. 145°.

Phenylhydrazone: m.p. 86°.

Di-Et acetal: b.p. 257°, 136–8°/16 mm. D<sub>4</sub><sup>20</sup> 1.0183. n<sub>D</sub><sup>20</sup> 1.4878.Rotbart, *Ann. chim.*, 1934, **1**, 479.**Phenoxyacetic Acid (Glycollic acid phenyl ether)**C<sub>6</sub>H<sub>5</sub>O·CH<sub>2</sub>·COOHC<sub>8</sub>H<sub>8</sub>O<sub>3</sub>

MW, 152

Needles from H<sub>2</sub>O. M.p. 98–9°. B.p. 285° decomp. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. *k* = 7.56 × 10<sup>-4</sup> at 25°.*o*-Phenylenediamine salt: m.p. 137°.*m*-Phenylenediamine salt: m.p. 125–6°.Di-*p*-phenylenediamine salt: m.p. 148–9° decomp.*Me* ester: C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>. MW, 166. B.p. 245°. D<sub>17.5</sub><sup>20</sup> 1.150.*Et* ester: C<sub>10</sub>H<sub>12</sub>O<sub>3</sub>. MW, 180. B.p. 251°, 145–50°/30 mm. D<sub>17.5</sub><sup>20</sup> 1.104.Phenyl ester: C<sub>14</sub>H<sub>12</sub>O<sub>3</sub>. MW, 228. M.p. 56°. B.p. 320–5°, 236°/73 mm., 185°/30 mm. (196°/13 mm.).Chloride: C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>Cl. MW, 170.5. B.p. 225–6°, 165°/60 mm., 111°/13 mm., 109°/9 mm.Amide: C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 151. Needles from H<sub>2</sub>O. M.p. 101–5°.Anhydride: C<sub>16</sub>H<sub>14</sub>O<sub>5</sub>. MW, 286. Leaflets from Et<sub>2</sub>O. M.p. 67–9°.

*Nitrile*:  $C_8H_7ON$ . MW, 133. B.p. 239–40°.  $D_{17}^{25}$  1.09.

*Anilide*: m.p. 101.5° (99°).

Monsanto, U.S.P., 1,974,810, (*Chem. Abstracts*, 1934, **28**, 7262).

van Alphen, *Rec. trav. chim.*, 1927, **46**, 144.

Blaise, Picard, *Ann. chim.*, 1912, **26**, 274.

**Phenoxyacetone** (*Phenyl acetonylether, acetol phenyl ether, ω-acetoanisole*)



$C_9H_{10}O_2$  MW, 150

Oil. B.p. 229–30°.

*Semicarbazone*: m.p. 173°.

*Guanyldiazotone*: m.p. 154°.

St. Wehln, *Ber.*, 1902, **35**, 3553 (*Foot-note*).

Stoermer, *Ber.*, 1895, **28**, 1253.

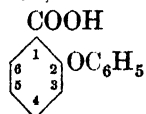
**Phenoxyanthraquinone.**

See under Hydroxyanthraquinone.

**4-Phenoxyazobenzene.**

See under 4-Hydroxyazobenzene.

**o-Phenoxybenzoic Acid** (*Salicylic acid phenyl ether, diphenyl ether 2-carboxylic acid*)



$C_{13}H_{10}O_3$  MW, 214

Leaflets from EtOH.Aq. M.p. 113°. B.p. 355°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. hot H<sub>2</sub>O. Dist. → trace of xanthone.

*Me ester*:  $C_{14}H_{12}O_3$ . MW, 228. B.p. 312°.

*Et ester*:  $C_{15}H_{14}O_3$ . MW, 242. B.p. above 360°.

*Phenyl ester*:  $C_{19}H_{16}O_3$ . MW, 290. Needles from EtOH. M.p. 109°.

*Amide*:  $C_{13}H_{11}O_2N$ . MW, 213. Prisms from EtOH. M.p. 131°. Sol. ord. org. solvents.

A.G.F.A., D.R.P., 158,998, (*Chem. Zentr.*, 1905, I, 843).

Ullmann, Zlokasoff, *Ber.*, 1905, **38**, 2117.

**m-Phenoxybenzoic Acid** (*m-Hydroxybenzoic acid phenyl ether, diphenyl ether 3-carboxylic acid*).

Needles from EtOH.Aq. M.p. 145°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Griess, *Ber.*, 1888, **21**, 980.

**p-Phenoxybenzoic Acid** (*p-Hydroxybenzoic acid phenyl ether, diphenyl ether 4-carboxylic acid*).

Prisms from CHCl<sub>3</sub>. M.p. 159.5°. Sol. EtOH, Et<sub>2</sub>O.

*Phenyl ester*: cryst. from EtOH.Aq. M.p. 73–8°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

Klein, *J. prakt. Chem.*, 1883, **28**, 199.

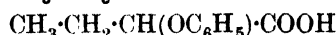
Griess, *Ber.*, 1888, **21**, 980.

Schönberg, Kraemer, *Ber.*, 1922, **55**, 1190.

**2-p-Phenoxybenzoylbenzoic Acid.**

See under 4'-Hydroxybenzophenone-2-carboxylic Acid.

**1-Phenoxybutyric Acid**



$C_{10}H_{12}O_3$  MW, 180

Needles from H<sub>2</sub>O, plates from ligroin. M.p. 82–3° (99°). B.p. 258°. Sol. ord. org. solvents.  $k = 6.82 \times 10^{-4}$ .

*Ag salt*: m.p. 202° decomp.

*Et ester*:  $C_{12}H_{16}O_3$ . MW, 208. B.p. 250–1°/748 mm., 175–80°/41 mm. Sol. EtOH, Et<sub>2</sub>O.

*Phenyl ester*:  $C_{16}H_{16}O_3$ . MW, 256. Prisms from MeOH. M.p. 48–9°. B.p. 202–3°/25 mm.  $D_{16}^{25}$  1.136.

*Chloride*:  $C_{10}H_{11}O_2Cl$ . MW, 198.5. B.p. 128–31°/38 mm.

*Amide*:  $C_{10}H_{13}O_2N$ . MW, 179. M.p. 111° (123°). Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, hot EtOH.

*Nitrile*:  $C_{10}H_{11}ON$ . MW, 161. B.p. 228–30°/748 mm. decomp.

*Anilide*: m.p. 93–4°.

Bischoff, *Ber.*, 1900, **33**, 931; 1901, **34**, 1837.

**3-Phenoxybutyric Acid**



$C_{10}H_{12}O_3$  MW, 180

Leaflets from ligroin. M.p. 64–5° (60°). Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. CS<sub>2</sub>, ligroin. Insol. H<sub>2</sub>O.

*Et ester*: b.p. 170–3°/25 mm.  $D_{25}^{25}$  1.048.  $n_D^{25}$  1.491.

*Amide*: leaflets from EtOH.Aq. M.p. 80°.

*Nitrile*: needles. M.p. 45–6°. B.p. 287–9°/765 mm.

Bentley, Haworth, Perkin, *J. Chem. Soc.*, 1896, **69**, 168.

Putochin, *Ber.*, 1922, **55**, 2747.

Nair, Peacock, *J. Indian Chem. Soc.*, 1935, **12**, 318.

Marvel, Tanenbaum, *J. Am. Chem. Soc.*, 1922, **44**, 2647.

**α-Phenoxydi-1-naphthylmethane.**

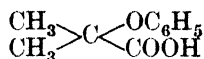
See under 1: 1'-Dinaphthylcarbinol.

**Phenoxy cyclohexane.**

See Phenyl cyclohexyl Ether.

**Phenoxyethyl Alcohol.**

See 2-Hydroxyethyl phenyl Ether.

**1-Phenoxyisobutyric Acid** $\text{C}_{10}\text{H}_{12}\text{O}_3$  MW, 180

Needles from  $\text{H}_2\text{O}$ . M.p.  $97-8^\circ$  ( $98-9^\circ$ ). Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , AcOH. Mod. sol. hot  $\text{H}_2\text{O}$ .  $k = 4.34 \times 10^{-4}$ . Volatile in steam.

Et ester:  $\text{C}_{12}\text{H}_{16}\text{O}_3$ . MW, 208. B.p.  $243^\circ$ ,  $127^\circ/12$  mm.

Phenyl ester:  $\text{C}_{16}\text{H}_{16}\text{O}_3$ . MW, 256. Needles. M.p.  $24-6^\circ$ . B.p.  $194-6^\circ/16$  mm.

Chloride:  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{Cl}$ . MW, 198.5. B.p.  $112-13^\circ/12.5$  mm. (impure).

Amide:  $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$ . MW, 179. Needles from EtOH. M.p.  $116^\circ$  ( $114^\circ$ ).

Bischoff, *Ber.*, 1900, **33**, 933; 1901, **34**, 1837.

Link, D.R.P., 80,986.

Gabriel, *Ber.*, 1913, **46**, 1347 (Footnote).

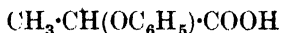
Bargellini, *Gazz. chim. ital.*, 1906, **36**, ii, 334.

**1 - Phenoxy naphthalene - 8 - carboxylic Acid.**

See under 8-Hydroxy-1-naphthoic Acid.

 **$\alpha$ -Phenoxyphenylacetic Acid.**

See under Mandelic Acid.

**1-Phenoxypropionic Acid (Lactic acid phenyl ether)** $\text{C}_9\text{H}_{10}\text{O}_3$  MW, 166

Needles from  $\text{H}_2\text{O}$ . M.p.  $115-16^\circ$  ( $112-13^\circ$ ). B.p.  $265-6^\circ/758$  mm. Sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ .  $k = 7.75 \times 10^{-4}$ . Volatile in steam.

Et ester:  $\text{C}_{11}\text{H}_{14}\text{O}_3$ . MW, 194. B.p.  $243-4^\circ$ ,  $160-4^\circ/50$  mm.,  $120-5^\circ/6$  mm.  $D_{17}^{25}$  1.360.

Di-methylaminoethyl ester:  $\text{C}_{13}\text{H}_{19}\text{O}_3\text{N}$ . MW, 237. M.p.  $75^\circ$ . B.p.  $116^\circ/0.2$  mm. Picrate: m.p.  $113^\circ$ . Picrolonate: m.p.  $148^\circ$ .

Phenyl ester:  $\text{C}_{16}\text{H}_{14}\text{O}_3$ . MW, 242. M.p.  $52^\circ$ . B.p.  $190^\circ/18$  mm.  $D_{15}^{25}$  1.147.

Tropine ester:  $\text{C}_{17}\text{H}_{23}\text{O}_3\text{N}$ . MW, 289. Yellow oil. B.p.  $149-50^\circ/0.6$  mm. Picrate: m.p.  $175^\circ$ . Picrolonate: m.p.  $200-2^\circ$ .

Chloride:  $\text{C}_9\text{H}_9\text{O}_2\text{Cl}$ . MW, 184.5. B.p.  $146-7^\circ/55$  mm.,  $120^\circ/30$  mm.,  $115^\circ/20$  mm., ( $115-17^\circ/10$  mm.).

Amide:  $\text{C}_9\text{H}_{11}\text{O}_2\text{N}$ . MW, 165. Needles from  $\text{H}_2\text{O}$ . M.p.  $132-3^\circ$  ( $130^\circ$ ). Sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH,  $\text{CS}_2$ , hot ligroin.

Diphenylamide: m.p.  $93^\circ$ .

Anilide: m.p.  $117^\circ$  ( $118-19^\circ$ ). B.p.  $211-12^\circ/14$  mm.

Me-anilide: m.p.  $57.5^\circ$ . B.p.  $206^\circ/18$  mm.

Et-anilide: m.p.  $47.5^\circ$ . B.p.  $224-5^\circ/17$  mm.

o-Nitroanilide: m.p.  $88^\circ$ . B.p.  $248^\circ/28$  mm.

m-Nitroanilide: m.p.  $118^\circ$ .

p-Nitroanilide: m.p.  $141-2^\circ$ .

o-Toluidide: m.p.  $88-90^\circ$ . B.p.  $220-30^\circ/24$  mm.

m-Toluidide: m.p.  $86.5^\circ$ . B.p.  $220^\circ/15$  mm.

p-Toluidide: m.p.  $115^\circ$ .

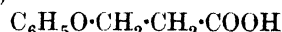
Phenylbenzylamide: m.p.  $111-12^\circ$ .

1-Naphthylamide: m.p.  $131^\circ$ . B.p.  $260^\circ/20$  mm.

2-Naphthylamide: m.p.  $117^\circ$ .

Plazek, Rodewald, Krzyzaniak, *Chem. Zentr.*, 1936, I, 1212.

Fourneau, Sandulesco, *Bull. soc. chim.*, 1922, **31**, 988.

**2-Phenoxypropionic Acid (Hydracrylic acid phenyl ether)** $\text{C}_9\text{H}_{10}\text{O}_3$  MW, 166

M.p.  $97.5-98^\circ$ . B.p.  $235-45^\circ/771$  mm.  $k = 5.4 \times 10^{-5}$ .

Powell, *J. Am. Chem. Soc.*, 1923, **45**, 2708.

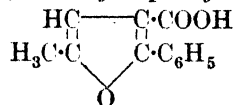
Bischoff, *Ber.*, 1900, **33**, 928.

**Phenoxypropylene oxide.**

See under Glycide.

**Phenthiazine.**

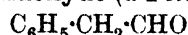
See Thiodiphenylamine.

**Phenuvic Acid (5-Methyl-2-phenylfuran-3-carboxylic acid, 5-methyl-2-phenyl- $\beta$ -furoic acid)** $\text{C}_{12}\text{H}_{10}\text{O}_3$  MW, 202

Needles from EtOH.Aq. M.p.  $147-8^\circ$  ( $144-5^\circ$ ). Sol. EtOH,  $\text{C}_6\text{H}_6$ , ligroin. Spar. sol. hot  $\text{H}_2\text{O}$ .

Et ester:  $\text{C}_{14}\text{H}_{14}\text{O}_3$ . MW, 230. Oil. B.p.  $193-4^\circ/20$  mm.

Borsche, Fels, *Ber.*, 1906, **39**, 1923, 1927.

**Phenylacetaldehyde ( $\alpha$ -Toluic aldehyde)** $\text{C}_8\text{H}_8\text{O}$  MW, 120

Cryst. from  $\text{H}_2\text{O}$ . M.p.  $33-4^\circ$ . B.p.  $195^\circ$ ,  $88^\circ/18$  mm.,  $78^\circ/10$  mm.  $D_{15}^{25}$  1.0272.  $n_D^{19}$  1.5255. Cold 10% KOH  $\rightarrow$  dimer.

Dimer: amorph. M.p.  $50^\circ$ . Sol. most org. solvents. Depolymerises at  $100^\circ$ .



*Cyanhydrin*: see under  $\alpha$ -Hydroxyhydrocinamic Acid.

*Di-Me acetal*: oil. B.p. 219–21°/754 mm.  $D_{20}^{25}$  1.0042.

*Di-Et acetal*: b.p. 245–6°.

*Oxime*: m.p. 98.5°.

*Phenylhydrazone*: prisms from ligroin. M.p. 62–3°.

*Diphenylhydrazone*: cryst. from EtOH. M.p. 101–2°.

2:4-Dinitrophenylhydrazone: golden leaflets from EtOH. M.p. 110°.

Erlenmeyer, Lipp, *Ann.*, 1883, **219**, 182.

Rassow, Burmeister, *J. prakt. Chem.*, 1911, **84**, 487.

Wood, Cowley, *J. Soc. Chem. Ind.*, 1923, **42**, 429r.

Kodama, *Chem. Abstracts*, 1922, **16**, 106.

### N-Phenylacetanilide.

See under Diphenylamine.

### Phenyl acetate



$\text{C}_8\text{H}_8\text{O}_2$  MW, 136

B.p. 195.7°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O.  $D_4^{20}$  1.0927,  $D_{16}^{25}$  1.0809.

Hoeflake, *Rec. trav. chim.*, 1916, **36**, 30.

### N-Phenylacethydrazide.

See Acetylphenylhydrazine.

**Phenylacetic Acid** ( $\alpha$ -Toluic acid, toluyllic acid)



$\text{C}_8\text{H}_8\text{O}_2$  MW, 136

Plates from pet. ether. M.p. 76–76.5°. B.p. 265.5°, 144.2–144.8°/12 mm.  $D_4^{27}$  1.091. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O.  $k = 5.56 \times 10^{-5}$  at 25°. Heat of comb.  $C_7$  930.7 Cal.

*Me ester*:  $\text{C}_9\text{H}_{10}\text{O}_2$ . MW, 150. B.p. 215°, 131–2°/50 mm.  $D_0^{20}$  1.0808,  $D_{16}^{25}$  1.0633.  $n_D^{25}$  1.5091.

*Et ester*:  $\text{C}_{10}\text{H}_{12}\text{O}_2$ . MW, 164. B.p. 227.1–227.6°, 132–8°/32 mm., 120–5°/17–18 mm.  $D_4^{20}$  1.0333.  $n_D^{25}$  1.49921. Used in perfumery.

*Propyl ester*:  $\text{C}_{11}\text{H}_{14}\text{O}_2$ . MW, 178. B.p. 238°.  $D^{20}$  1.0142.

*Isobutyl ester*:  $\text{C}_{12}\text{H}_{16}\text{O}_2$ . MW, 192. B.p. 247°.

*tert.-Butyl ester*: f.p. – 21°. B.p. 110°/15 mm.  $D_4^{20}$  0.9758.  $n_D$  1.4825.

*d-Amyl ester*: b.p. 265–6°/722.7 mm.  $D_4^{20}$  0.982.  $n_D^{21}$  1.4872.  $[\alpha]_D^{25} + 4.92^\circ$ .

*l-Menthyl ester*: liq. B.p. 216°/39 mm., 197°/15 mm., 94–5°/0.25 mm.  $D_4^{20}$  0.9874.  $[\alpha]_D^{25} - 68.15^\circ$ .

*Phenyl ester*:  $\text{C}_{14}\text{H}_{12}\text{O}_2$ . MW, 212. Needles from EtOH.Aq. M.p. 42°. B.p. 158°/7 mm.

Sol. EtOH, AcOH, pet. ether. Spar. sol. cold Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Benzyl ester*:  $\text{C}_{15}\text{H}_{14}\text{O}_2$ . MW, 226. Oil. B.p. 317–19°, 270°/160 mm., 175–6°/12 mm.  $D^{17}$  1.0938. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O.

*p-Nitrobenzyl ester*: m.p. 65°.

*p-Tolyl ester*: m.p. 75–6°.

*Fluoride*: phenacetyl fluoride.  $\text{C}_8\text{H}_7\text{OF}$ . MW, 138. Oil. B.p. 88–9°/17 mm.

*Chloride*: phenacetyl chloride.  $\text{C}_8\text{H}_7\text{OCl}$ . MW, 154.5. Liq. B.p. 170°/250 mm., 104–5°/24 mm., 100°/12 mm.  $D_4^{20}$  1.1856,  $D_{16}^{25}$  1.16817.

*Bromide*: phenacetyl bromide.  $\text{C}_8\text{H}_7\text{OBr}$ . MW, 199. B.p. 150–5°/50 mm.

*Amide*: phenacetamide.  $\text{C}_8\text{H}_9\text{ON}$ . MW, 135. Plates or leaflets. M.p. 157°. Sol. EtOH.

Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ . *N-Acetyl*: needles from EtOH.Aq. M.p. 129°. *N-Benzoyl*: prisms from  $\text{C}_6\text{H}_6$ , needles from EtOH. M.p. 129–30°.

*Methylamide*: cryst. from  $\text{C}_6\text{H}_6$ . M.p. 58°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O,  $\text{C}_6\text{H}_6$ .

*Dimethylamide*: cryst. M.p. 43.5°. B.p. 155°/10 mm. Sol. H<sub>2</sub>O, Et<sub>2</sub>O, CHCl<sub>3</sub>,  $\text{C}_6\text{H}_6$ .

*Ethylamide*: plates from H<sub>2</sub>O. M.p. 73–4°.

*Diethylamide*: leaflets. M.p. 86°. B.p. 297°.

*Dipropylamide*: oil. B.p. 183–4°/16 mm.

*Nitrile*: see Benzyl cyanide.

*Anhydride*:  $\text{C}_{16}\text{H}_{14}\text{O}_3$ . MW, 254. Needles. M.p. 72°.

*Anilide*: prisms from EtOH. M.p. 117–18°. Sol. EtOH, Et<sub>2</sub>O.

*o-Toluidide*: needles from  $\text{C}_6\text{H}_6$ . M.p. 159°. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>,  $\text{C}_6\text{H}_6$ . Spar. sol. Et<sub>2</sub>O, CS<sub>2</sub>, ligroin.

*p-Toluidide*: leaflets from EtOH. M.p. 135–6°. Mod. sol. EtOH, Et<sub>2</sub>O.

*Hydrazide*: needles from H<sub>2</sub>O. M.p. 116°. Sol. warm EtOH, H<sub>2</sub>O. Mod. sol. hot Et<sub>2</sub>O. Reduces warm Fehling's. *B.HCl*: needles. M.p. 215° decomp. Sol. H<sub>2</sub>O.

Adams, Thal, *Organic Syntheses*, Collective Vol. I, 265, 427.

Heilbron, Hey, Lythgoe, *J. Chem. Soc.*, 1936, 297.

Sobin, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 2458.

Yabroff, Porter, *J. Am. Chem. Soc.*, 1932, **54**, 2453.

Kindler, Giese, Hesse, *Arch. Pharm.*, 1927, **265**, 389.

Wieland, Fischer, *Ann.*, 1925, **446**, 65.

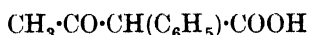
Autenrieth, Thomae, *Ber.*, 1924, **57**, 431.

Adams, Ulich, *J. Am. Chem. Soc.*, 1920, **42**, 599.

Traube, Krahmer, *Ber.*, 1919, **52**, 1296.

Taverne, *Rec. trav. chim.*, 1897, **16**, 34.

## 1-Phenylacetoacetic Acid


 $\text{C}_{10}\text{H}_{10}\text{O}_3$  MW, 178

*Et ester*:  $\text{C}_{12}\text{H}_{14}\text{O}_3$ . MW, 206. Oil. B.p.  $145-7^\circ/11$  mm. Mod. sol. alkalis. Alc.  $\text{FeCl}_3 \rightarrow$  intense violet col.

*1-Menthyl ester*: needles from MeOH. M.p.  $69^\circ$ . B.p.  $204-10^\circ/10$  mm.,  $131-3^\circ/0.1$  mm. Shows mutarotation.

*Nitrile*:  $\text{C}_{10}\text{H}_9\text{ON}$ . MW, 159. Cryst. from EtOH.Aq. or AcOEt-pet. ether. M.p.  $90-1^\circ$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. hot  $\text{H}_2\text{O}$ , pet. ether. Volatile in steam. Alc.  $\text{FeCl}_3 \rightarrow$  green col. *Phenylhydrazone*: needles from MeOH. M.p.  $114^\circ$ . *Anil*: leaflets from  $\text{C}_6\text{H}_6$ . M.p.  $102-3^\circ$ .

*Anilide*: needles from EtOH.Aq. M.p.  $97^\circ$ . Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ .

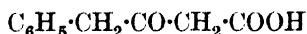
Rupe, *Ann.*, 1913, **398**, 372.

Bodroux, *Bull. soc. chim.*, 1910, **7**, 851.

Beckh, *Ber.*, 1898, **31**, 3161.

Heller, Herrmann, Spielmeyer, *J. prakt. Chem.*, 1928, **120**, 196.

## 3-Phenylacetoacetic Acid


 $\text{C}_{10}\text{H}_{10}\text{O}_3$  MW, 178

*Et ester*:  $\text{C}_{12}\text{H}_{14}\text{O}_3$ . MW, 206. B.p.  $153-5^\circ/9$  mm. MeOH +  $\text{FeCl}_3 \rightarrow$  deep red col. Tetranitromethane in MeOH  $\rightarrow$  faint yellowish-green col., without solvent  $\rightarrow$  reddish-orange col. Does not reduce Fehling's.  $\text{NaNO}_2 \rightarrow$  deep blue col.

*Cu salt*: cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $176-8^\circ$ . Sol.  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ .

*Semicarbazone*: needles from MeOH.Aq. M.p.  $113-16^\circ$ . Sol. EtOH. Insol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ .

*Anil*: needles from 75% MeOH. M.p.  $96-98.5^\circ$ . Sol.  $\text{Et}_2\text{O}$ , AcOEt. Spar. sol. cold MeOH, pet. ether, ligroin. Insol.  $\text{H}_2\text{O}$ .

Sonn, Litten, *Ber.*, 1933, **66**, 1512.

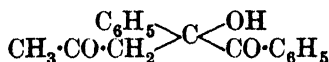
## Phenylacetol.

See 3-Phenylhydroxyacetone.

## Phenylacetone.

See Methyl benzyl Ketone.

**Phenylacetylbenzoylcarbinol** (*Acetone-benzil*)


 $\text{C}_{17}\text{H}_{16}\text{O}_3$  MW, 268

Prisms from  $\text{Et}_2\text{O}$ . M.p.  $78^\circ$ . Sol.  $\text{Et}_2\text{O}$ , hot EtOH. At  $200^\circ \rightarrow$  benzil + acetone. Ox.  $\rightarrow$  acetic + benzoic acids.

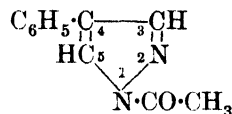
*Monoxime*: cryst. from EtOH. M.p.  $146^\circ$ . Sol. hot  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{Et}_2\text{O}$ .

Japp, Miller, *J. Chem. Soc.*, 1885, **47**, 24.

## Phenyl acetonyl Ether.

See Phenoxyacetone.

**4-Phenyl-1-acetopyrazole** (*Acetylphenylpyrazole*)


 $\text{C}_{11}\text{H}_{10}\text{ON}_2$  MW, 186

Pale yellow needles from MeOH or  $\text{Et}_2\text{O}$ . M.p.  $81.5-82.5^\circ$ . B.p.  $159-61^\circ/12$  mm.  $D_4^{20}$  1.0945.  $n_D$  1.5589.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 177.

**1-Phenyl-3-acetopyrazole** (*3-Acetyl-1-phenylpyrazole*).

*Phenylhydrazone*: yellow plates. M.p.  $182^\circ$ .

Diels, Petersen, *Ber.*, 1922, **55**, 3454.

**1-Phenyl-4-acetopyrazole** (*4-Acetyl-1-phenylpyrazole*).

Needles from EtOH.Aq. M.p.  $121.5-122.5^\circ$ . Sol.  $\text{H}_2\text{O}$ .

*Oxime*: needles from 50% EtOH. M.p.  $129-31^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ .

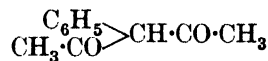
*Phenylhydrazone*: needles from EtOH. M.p.  $142-4^\circ$  decomp.

Balbiano, *Gazz. chim. ital.*, 1889, **19**, 197.

## N-Phenylaceturic Acid.

See under Phenylglycine.

**Phenylacetylacetone** (*3-Phenylpentandione-2:4*)

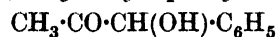

 $\text{C}_{11}\text{H}_{12}\text{O}_2$  MW, 176

Oil with odour of pine shavings. M.p.  $58-60^\circ$ . B.p.  $127-8^\circ/15$  mm.  $\text{FeCl}_3 \rightarrow$  purple col.

*Cu salt*: dark green cryst. Decomp. at  $222-4^\circ$ . Sol. usual org. solvents.

Morgan, Drew, Porter, *Ber.*, 1925, **58**, 333.

**Phenylacetylcarbinol** (*Methyl  $\alpha$ -hydroxybenzyl ketone, 1-hydroxy-1-phenylacetone*)


 $\text{C}_9\text{H}_{10}\text{O}_2$  MW, 150

Yellowish oil. B.p.  $205-7^\circ$ ,  $135-7^\circ/24$  mm.,  $140-5^\circ/11$  mm. Misc. with most org. solvents. Reduces  $\text{NH}_3 \cdot \text{AgNO}_3$  and Fehling's.

*Acetyl*: pale yellow oil. B.p.  $165-70^\circ/40$  mm.

*Oxime*: needles from  $\text{H}_2\text{O}$ . M.p.  $112-15^\circ$ .

**Semicarbazone:** powder from toluene. M.p. 194°. Sol. boiling AcOH. Spar. sol. other solvents.

**2:4-Dinitrophenylhydrazone:** orange needles from EtOH. M.p. 170°.

**Note:** phenylacetylcarbinol is tautomeric (keto-carbinol tautomerism) with methylbenzoylcarbinol ( $\beta$ -hydroxypropioophenone).

Hey, *J. Chem. Soc.*, 1930, 1232.

Meerwein, Hinz, *Ann.*, 1930, 484, 1.

Favorskiĭ, Tennikova, *Compt. rend.*, 1934, 198, 1998.

Freudenberg, *Biochem. Z.*, 1932, 245, 238.

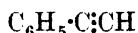
Roger, *Biochem. Z.*, 1931, 230, 320.

Kotchergine, *Bull. soc. chim.*, 1928, 43, 573.

### Phenylacetyl-carbinol.

See 3-Phenylhydroxyacetone.

**Phenylacetylene** (*Acetylenylbenzene, ethinylbenzene*)



$\text{C}_8\text{H}_6$  MW, 102

Oil. F.p.  $-45^\circ$ . B.p.  $142-4^\circ$ ,  $49-50^\circ/14$  mm.  $D_4^{25}$  0.9371.  $n_D^{25}$  1.5501. Heat of comb.  $\text{C}_v$  1024.5 Cal.

Vaughn, *J. Am. Chem. Soc.*, 1934, 56, 2064.

Hessler, *Organic Syntheses*, Collective Vol. I, 428.

### Phenylacetylene dichloride.

See  $\alpha\beta$ -Dichlorostyrene.

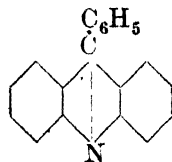
### Phenylacetylglycine.

See Phenaceturic Acid.

### 2-Phenylacetyl-propionic Acid.

See 4-Phenyl-levulinic Acid.

### 5-Phenylacridine (ms-Phenylacridine)



$\text{C}_{19}\text{H}_{13}\text{N}$

MW, 255

Pale yellow leaflets from EtOH. M.p.  $184^\circ$  ( $181^\circ$ ). B.p.  $403-4^\circ$ . Sol.  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{Et}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{H}_2\text{O}$ . Sol. conc.  $\text{H}_2\text{SO}_4$ . Sols. in acids show green fluor. Sublimes in leaflets. Triboluminescent.

**Picrate:** cryst. from EtOH. M.p.  $227.7^\circ$ .

**Dipicrate:** cryst. from EtOH. M.p.  $185-6^\circ$ .

**Methohydroxide:** plates from  $\text{C}_6\text{H}_6$ . M.p.  $140^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether. Insol.  $\text{H}_2\text{O}$ . Sol. acids.

**Methochloride:** cryst. +  $2\text{H}_2\text{O}$ . Decomp. at  $225-6^\circ$ . Very sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{CHCl}_3$ , Py,  $\text{PhNO}_2$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CCl}_4$ .

**Methobromide:** greenish-yellow cryst. +  $\text{H}_2\text{O}$ . Decomp. at  $230^\circ$ . Sol.  $\text{H}_2\text{O}$ . Mod. sol. EtOH,  $\text{CHCl}_3$ , Py,  $\text{PhNO}_2$ , aniline.

**Methiodide:** black prisms from EtOH. Sol. EtOH,  $\text{CHCl}_3$ , Py,  $\text{PhNO}_2$ . Less sol.  $\text{H}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CCl}_4$ .

**Methonitrate:** yellow needles. Decomp. at  $288^\circ$ . Sol. EtOH,  $\text{CHCl}_3$ .

**Ethohydroxide:** cryst. from toluene. M.p.  $136-7^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ .

**Ethiodide:** dark red needles from  $\text{H}_2\text{O}$ . M.p.  $223^\circ$  decomp.

**Ethopicrate:** yellow needles from EtOH. M.p.  $181^\circ$ .

**Phenohydroxide:** prisms from ligroin. M.p.  $178^\circ$ . Sol.  $\text{Et}_2\text{O}$ , EtOH,  $\text{C}_6\text{H}_6$ , hot ligroin. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with intense green fluor.

Ullmann, Maag, *Ber.*, 1907, 40, 2520.

Schmid, Decker, *Ber.*, 1906, 39, 937.

Kaufmann, Albertini, *Ber.*, 1909, 42, 2008.

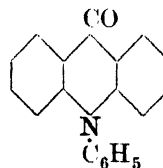
Bernthsen, *Ann.*, 1884, 224, 13.

Ullmann, *Ann.*, 1907, 355, 319.

### 4-Phenylacridinic Acid.

See 4-Phenylquinoline-2:3-dicarboxylic Acid.

### N-Phenylacridone



$\text{C}_{19}\text{H}_{13}\text{ON}$

MW, 271

Yellow cryst. from toluene. M.p.  $276^\circ$ . Sol. boiling  $\text{C}_6\text{H}_6$ , amyl alcohol, toluene with yellow col. Mod. sol. AcOH with intense blue fluor. Spar. sol. MeOH, EtOH. Insol.  $\text{Et}_2\text{O}$ , ligroin. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with intense green fluor.

Goldberg, Nimcrovsky, *Ber.*, 1907, 40, 2450.

### 2-Phenylacrolein.

See Cinnamaldehyde.

### 1-Phenylacrylic Acid.

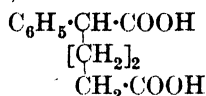
See Atropic Acid.

### 2-Phenylacrylic Acid.

See Cinnamic Acid.

## 1-Phenyladipic Acid

**1-Phenyladipic Acid** (1-Phenylbutane-1 : 4-dicarboxylic acid)

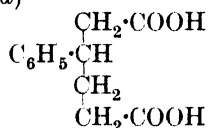


$\text{C}_{12}\text{H}_{14}\text{O}_4$  MW, 222

Cryst. from  $\text{C}_6\text{H}_6\text{-Et}_2\text{O}$ . M.p. 132-3°.

Case, *J. Am. Chem. Soc.*, 1933, **55**, 2927.

**2-Phenyladipic Acid** (2-Phenylbutane-1 : 4-dicarboxylic acid)



$\text{C}_{12}\text{H}_{14}\text{O}_4$  MW, 222

Plates from  $\text{C}_6\text{H}_6\text{-Et}_2\text{O}$ . M.p. 148°. Sol. EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

Di-Et ester :  $\text{C}_{16}\text{H}_{21}\text{O}_4$ . MW, 277. B.p. 197-200°/10 mm.

v. Braun, Weissbach, *Ber.*, 1931, **64**, 1785.

Planske, *J. Am. Chem. Soc.*, 1931, **53**, 1104.

### Phenylalanine.

See Aminohydrocinnamic Acid and Anilino-propionic Acid.

### Phenylaldehydoacetic Acid.

See Phenylformylacetic Acid.

**1-Phenylallyl Alcohol** (Vinylphenylcarbinol,  $\alpha$ -hydroxyallylbenzene)



$\text{C}_9\text{H}_{10}\text{O}$  MW, 134

Mobile oil. B.p. 214°/760 mm., 114°/25 mm., 106°/18 mm., 90-2°/11 mm. Decolourises  $\text{KMnO}_4$ .  $\text{O}_3 \rightarrow$  mandelic acid + formic acid. Sweet taste. Odourless.

Et ether :  $\text{C}_{11}\text{H}_{14}\text{O}$ . MW, 162. Oil. B.p. 203-5°/755 mm. Fruity odour.

Benzoyl : mobile oil. B.p. 182°/12 mm.

p-Nitrobenzoyl : leaflets from EtOH. M.p. 48°.

Klages, Klenk, *Ber.*, 1906, **39**, 2553.

Rupe, Müller, *Helv. Chim. Acta*, 1921, **4**, 846.

Moureu, Gallagher, *Bull. soc. chim.*, 1921, **29**, 1009.

Meisenheimer, Schmidt, *Ann.*, 1929, **475**, 178.

### 3-Phenylallyl Alcohol.

See Cinnamyl Alcohol.

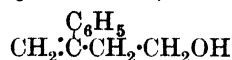
### Phenylallylamine.

See Allylaniline.

379

## N-Phenyl-2-amino-8-naphthol-6-sulphonic Acid

**2-Phenylallylcarbinol** (2-Phenyl-1-butenol-4)



$\text{C}_{10}\text{H}_{12}\text{O}$  MW, 148

B.p. 123°/10 mm.  $D^{20}$  1.0272.  $n_D^{20}$  1.5577.

St. Pfau, Plattner, *Helv. Chim. Acta*, 1932, **15**, 1266.

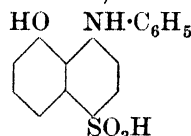
### Phenylallylene.

See Methylphenylacetylene.

### Phenyl o-aminobenzyl Ether.

See under o-Aminobenzyl Alcohol.

**N-Phenyl-1-amino-8-naphthol-4-sulphonic Acid** (Phenyl-S acid, 8-anilino-1-naphthol-5-sulphonic acid)



$\text{C}_{16}\text{H}_{13}\text{O}_4\text{NS}$  MW, 315

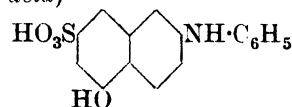
Cryst. Intermediate for azo dyes.

Acid Na salt : needles. Mod. sol.  $\text{H}_2\text{O}$ .

Neutral Na salt : cryst. Mod. sol. cold  $\text{H}_2\text{O}$ .

Bayer, D.R.P., 181,929, (*Chem. Zentr.*, 1907, **I**, 1653).

**N-Phenyl-2-amino-5-naphthol-7-sulphonic Acid** (Phenyl-J acid, 2-anilino-5-naphthol-7-sulphonic acid, 6-anilino-1-naphthol-3-sulphonic acid)



$\text{C}_{16}\text{H}_{13}\text{O}_4\text{NS}$  MW, 315

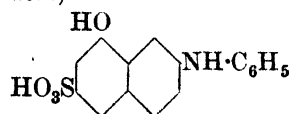
Cryst. Spar. sol.  $\text{H}_2\text{O}$ , EtOH, cold dil. acids. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  green sol. Dil.  $\text{Na}_2\text{CO}_3 \rightarrow$  sol. with violet fluor. Important intermediate for azo dyes.

Leonhardt, D.R.P., 114,248, (*Chem. Zentr.*, 1900, **II**, 997).

Badische, D.R.P., 122,570, (*Chem. Zentr.*, 1901, **II**, 670).

Bucherer, Stohmann, *J. prakt. Chem.*, 1905, **71**, 451.

**N-Phenyl-2-amino-8-naphthol-6-sulphonic Acid** (Phenyl-gamma acid, 2-anilino-8-naphthol-6-sulphonic acid, 7-anilino-1-naphthol-3-sulphonic acid)



$\text{C}_{16}\text{H}_{13}\text{O}_4\text{NS}$  MW, 315

Needles or leaflets. Sol. 35 parts hot, 300 parts cold  $H_2O$ . Intermediate for azo dyes.

*Ba salt*: leaflets. Mod. sol.  $H_2O$ .

See last two references above and also

Cassella, D.R.P., 79,014.

Levinstein, D.R.P., 99,339, (*Chem. Zentr.*, 1899, I, 160).

### Phenyl-aminonaphthyl-methane.

See 4-Benzyl-1-naphthylamine.

**1-Phenyl-*n*-amyl Alcohol** (1-Phenyl-pentanol-1, butylphenylcarbinol,  $\alpha$ -hydroxy-*n*-amylbenzene)



$C_{11}H_{16}O$  MW, 164

B.p. 137°/21 mm., 132°/14 mm., 121–2°/15 mm.

*Urethane*: m.p. 75°.

Levene, Mikeska, *J. Biol. Chem.*, 1926, 70, 355.

Fourneau, Montaigne, Puyal, *Chem. Abstracts*, 1922, 16, 240.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, 57, 155.

**3-Phenyl-*n*-amyl Alcohol** (3-Phenyl-pentanol-1)



$C_{11}H_{16}O$  MW, 164

*l.*

B.p. 135–8°/20 mm., 118°/1 mm.  $[\alpha]_D^{25} - 2.69^\circ$ .

Levene, Marker, *J. Biol. Chem.*, 1935, 110, 333.

Lévy, *Compt. rend.*, 1933, 197, 773.

**4-Phenyl-*n*-amyl Alcohol** (4-Phenyl-pentanol-1)



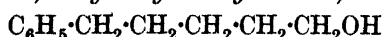
$C_{11}H_{16}O$  MW, 164

*l.*

B.p. 109°/1 mm.  $[\alpha]_D^{25} - 0.90^\circ$ .

Levene, Marker, *J. Biol. Chem.*, 1935, 110, 339.

**5-Phenyl-*n*-amyl Alcohol** (5-Phenyl-pentanol-1,  $\omega$ -hydroxy-*n*-amylbenzene)



$C_{11}H_{16}O$  MW, 164

B.p. 155°/20 mm., 151°/13 mm.

*Phenyl ether*:  $C_{17}H_{20}O$ . MW, 240. Viscous liq. B.p. 198°/14 mm.

*Acetyl*: b.p. 155°/12 mm.

v. Braun, *Ber.*, 1911, 44, 2872.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, 57, 155.

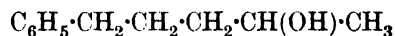
### 1-Phenyl-*sec*.-*n*-amyl Alcohol.

See Propylbenzylcarbinol.

### 2-Phenyl-*sec*.-*n*-amyl Alcohol.

See Methylpropylphenylcarbinol.

**5-Phenyl-*sec*.-*n*-amyl Alcohol** (5-Phenyl-pentanol-2,  $\delta$ -hydroxy-*n*-amylbenzene, 1-phenyl-pentanol-4)



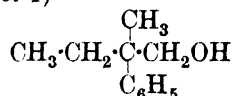
$C_{11}H_{16}O$  MW, 164

B.p. 134–5°/16 mm.  $D_4^{25} 0.9643$ .  $n_{D}^{25} 1.51110$ .

*Phenylurethane*: needles from pet. ether. M.p. 57°.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, 57, 151.

**2-Phenyl-active-amyl Alcohol** (2-Methyl-2-phenylbutanol-1)



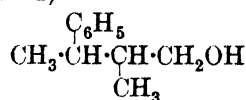
$C_{11}H_{16}O$  MW, 164

Oil. B.p. 246°, 138°/23 mm.

*Benzoyl*: cryst. M.p. 46°. B.p. 202–4°/12 mm.

Blondeau, *Compt. rend.*, 1922, 174, 1424.

**3-Phenyl-active-amyl Alcohol** (2-Methyl-3-phenylbutanol-1)

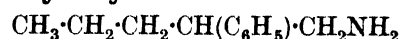


$C_{11}H_{16}O$  MW, 164

Oil with odour of roses. B.p. 132–3°/13 mm.

Braun *et al.*, *Ann.*, 1927, 451, 48.

### 2-Phenylamylamine



$C_{11}H_{17}N$  MW, 163

*l.*

Liq. B.p. 90°/3 mm.

Levene, Marker, *J. Biol. Chem.*, 1931, 93, 773.

**5-Phenyl-*n*-amylamine** ( $\omega$ -Amino-*n*-amylbenzene)



$C_{11}H_{17}N$  MW, 163

Liq. B.p. 131°/15 mm.

*N-Di-Me*:  $C_{13}H_{21}N$ . MW, 191. B.p. 134–5°/18 mm.

*N-Et*:  $C_{13}H_{21}N$ . MW, 191. B.p. 295°. *B,HCl*: m.p. 144°.

*N*-Amyl:  $C_{16}H_{27}N$ . MW, 233. B.p. 310°. *B.HCl*: cryst. from  $H_2O$  or EtOH. M.p. 184–6°.

*N*-Cyclohexyl:  $C_{17}H_{28}N$ . MW, 246. B.p. 315°. *B.HCl*: m.p. 246°.

*Benzoyl*: needles from  $Et_2O$ -ligroin. M.p. 60°. B.p. 273–5°/15 mm.

$B_2H_2PtCl_6$ : leaflets from  $H_2O$ . M.p. 220°.

*Picrate*: cryst. from  $Et_2O$ . M.p. 152–3°. Sol. EtOH.

*Methiodide*: leaflets from  $H_2O$ . M.p. 181°. Very sol.  $CHCl_3$ . Spar. sol.  $H_2O$ , EtOH.

*Methochloroplatinate*: light red cryst. from  $H_2O$ . M.p. 219°.

Skita, Wulff, *Ann.*, 1927, **455**, 40.

v. Braun, *Ber.*, 1910, **43**, 2849.

Merck, D.R.P., 238,959, (*Chem. Zentr.*, 1911, II, 1284).

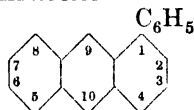
### Phenylamylene.

See Phenylpentene.

### *N*-Phenylanisidine.

See under 2- and 4-Hydroxydiphenylamines.

### 1-Phenylanthracene



$C_{20}H_{14}$

MW, 254

Yellow prisms from EtOH. M.p. 110–12°. Sol. usual solvents.

Cook, *J. Chem. Soc.*, 1930, 1091.

### 2-Phenylanthracene.

Pale yellow leaflets from AcOEt. M.p. 207–207.5°. Spar. sol. most solvents.

See previous reference.

### 9-Phenylanthracene (ms-Phenylanthracene).

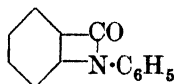
Leaflets from EtOH. M.p. 152–3°. B.p. 417°. Sol. warm EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $CHCl_3$ . Sols. show blue fluor.

Baeyer, *Ann.*, 1880, **202**, 61.

Linebarger, *Am. Chem. J.*, 1891, **13**, 554.

Guyot, Catel, *Compt. rend.*, 1905, **140**, 1462.

### *N*-Phenylanthranil



$C_{13}H_9ON$

MW, 195

Pale yellow needles from 50% EtOH. M.p. 169° decomp. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ , AcOEt,  $C_6H_6$ . Spar. sol.  $H_2O$ . Sols. show greenish-blue fluor.

*B.HCl*: pale yellow needles. M.p. 169° decomp.

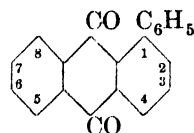
*B.HgCl\_2*: golden-yellow leaflets. M.p. 225–32° decomp.

Drechsler, *Monatsh.*, 1914, **35**, 533.

### Phenylanthranilic Acid.

See Diphenylamine-2-carboxylic Acid.

### 1-Phenylanthraquinone



$C_{20}H_{12}O_2$

MW, 284

Yellow needles from AcOH. M.p. 176–8°.

Allen, Overbaugh, *J. Am. Chem. Soc.*, 1935, **57**, 740.

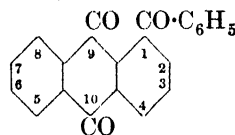
### 2-Phenylanthraquinone.

Pale yellow needles from MeOH. M.p. 162–5° (160–1°). Sol. AcOH,  $CCl_4$ . Mod. sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $Me_2CO$ ,  $CS_2$ ,  $C_6H_6$ . Alc. KOH  $\rightarrow$  red col. Conc.  $H_2SO_4 \rightarrow$  red col.

Scholl, Neovius, *Ber.*, 1911, **44**, 1079.

Groggins, *Ind. Eng. Chem.*, 1930, **22**, 620.

### Phenyl 1-anthraquinonyl Ketone (1-Benzoylanthraquinone)



$C_{21}H_{12}O_3$

MW, 312

Yellowish cryst. powder from AcOH. M.p. 229° (225–7°). Sol. conc.  $H_2SO_4$  with pale yellow col.

1-Oxime: yellow rhombic leaflets. Decomp. about 218°.

9-Oxime: *syn.*, yellow rhombic cryst. Decomp. about 210°. *Anti.*: yellow needles or leaflets. M.p. 230°.

Scholl, Müller, Stix, *Ber.*, 1935, **68**, 801.

Scholl *et al.*, *Ann.*, 1934, **512**, 1.

I.G., D.R.P., 487,254, (*Chem. Abstracts*, 1930, **24**, 2147).

Schaarschmidt, *Ber.*, 1915, **48**, 837.

### Phenylarsen-dibromide.

See Phenyldibromoarsine.

### Phenylarsen-dichloride.

See Phenyldichloroarsine.

### Phenylarsine (Arsinobenzene)

$C_6H_5 \cdot AsH_2$

$C_6H_7As$

MW, 154

Oil. B.p. 148°, 93°/70 mm., 77°/33 mm., 55°/14 mm., 36°/2 mm. Sol. EtOH, Et<sub>2</sub>O. In air → phenylarsinic acid.

As-Di-Me: C<sub>8</sub>H<sub>11</sub>As. MW, 182. Liq. B.p. 200°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. *Methiodide*: prisms from EtOH. M.p. 248.5° (244°). *Methochloroplatinate*: red plates from hot H<sub>2</sub>O. M.p. 219° decomp.

As-Di-Et: C<sub>10</sub>H<sub>15</sub>As. MW, 210. B.p. 240°. *Methiodide*: prisms from EtOH-Et<sub>2</sub>O. M.p. 122° (77°). *Ethiodide*: prisms from H<sub>2</sub>O. M.p. 112-13°. Sol. EtOH. Insol. Et<sub>2</sub>O.

As-Et-Propyl: C<sub>11</sub>H<sub>17</sub>As. MW, 224. B.p. 245°.

As-Phenyl: see Diphenylarsine.

As-Diphenyl: see Triphenylarsine.

Palmer, Dehn, *Ber.*, 1901, **34**, 3598.

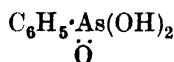
Fichter, Elkind, *Ber.*, 1916, **49**, 245.

Winmill, *J. Chem. Soc.*, 1912, **101**, 719.

Michaelis, Link, *Ann.*, 1881, **201**, 205.

La Coste, Michaelis, *Ann.*, 1880, **201**, 212.

### Phenylarsinic Acid (Phenylarsonic acid)



C<sub>6</sub>H<sub>7</sub>O<sub>3</sub>As MW, 202

Cryst. from H<sub>2</sub>O. M.p. 158-62° decomp. Above m.p. → anhydride. 3.25 parts sol. 100 parts H<sub>2</sub>O at 28°, 24 parts at 84°. Very stable to ox. agents.

*Anhydride*: amorph. Decomp. on heating.

Di-Me ester: C<sub>8</sub>H<sub>11</sub>O<sub>3</sub>As. MW, 230. B.p. 188°/95 mm. D<sub>20</sub> 1.3946. Decomp. by H<sub>2</sub>O.

Di-Et ester: C<sub>10</sub>H<sub>15</sub>O<sub>3</sub>As. MW, 258. B.p. 168-70°/15 mm. D<sub>15</sub> 1.318.

Dichloride: C<sub>6</sub>H<sub>5</sub>OCl<sub>2</sub>As. MW, 239. Cryst. M.p. about 100°. Sol. H<sub>2</sub>O with decomp.

La Coste, Michaelis, *Ann.*, 1880, **201**, 203.

Michaelis, Loesner, *Ber.*, 1884, **27**, 265.

Michaelis, *Ann.*, 1902, **320**, 293.

Bullard, Dickey, *Organic Syntheses*, 1935, **XV**, 59.

### Phenylarsonic Acid.

See Phenylarsinic Acid.

### N-Phenylaspartic anil.

See Anilinosuccinanyl.

**Phenylitaconic Acid** (*Iso-γ-phenylitaconic acid*)



C<sub>11</sub>H<sub>10</sub>O<sub>4</sub> MW, 206

Prisms from Et<sub>2</sub>O. M.p. 149-51° (not sharp). Sol. 15 parts Et<sub>2</sub>O, 90 parts H<sub>2</sub>O at ord. temps. Spar. sol. CHCl<sub>3</sub>. Insol. CS<sub>2</sub>, ligroin. Sunlight

→ phenylitaconic acid. Heat in vacuo at 160-75° → anhydride. KMnO<sub>4</sub> → benzaldehyde + malonic acid + oxalic acid.

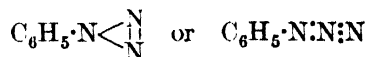
*Anhydride*: needles from Et<sub>2</sub>O-CHCl<sub>3</sub>. M.p. 138-40°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O.

Stobbe, Horn, *Ber.*, 1908, **41**, 3983.

Stobbe, *Ber.*, 1908, **41**, 4353.

Fittig, Brooke, *Ann.*, 1899, **305**, 33.

**Phenyl azide** (*Azidobenzene, diazobenzolimide, phenylazoimide, triazobenzene*)

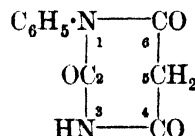


C<sub>6</sub>H<sub>5</sub>N<sub>3</sub> MW, 119

Pale yellow oil. B.p. 73.5°/22-4 mm., 59°/14 mm. Mod. sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. D<sub>4</sub> 1.2399, D<sub>20</sub> 1.0980, D<sub>25</sub> 1.0853. n<sub>D</sub><sup>20</sup> 1.56104. Possesses aromatic ammoniacal odour. Explodes on dist. at ord. press., or by treatment with conc. H<sub>2</sub>SO<sub>4</sub>. Volatile in steam. Strongly antipyretic. N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O → C<sub>6</sub>H<sub>6</sub> + NH<sub>3</sub> + N. NaHg + EtOH → hydrazobenzene. Zn + H<sub>2</sub>SO<sub>4</sub> → C<sub>6</sub>H<sub>6</sub> + NH<sub>3</sub> + N. Dil. H<sub>2</sub>SO<sub>4</sub> → *p*-aminophenol. Na + EtOH → aniline + N. Long heating with conc. HCl → *o*- and *p*-chloroanilines.

Dimroth, *Ber.*, 1902, **35**, 1032 (Note).

### 1-Phenylbarbituric Acid



C<sub>10</sub>H<sub>8</sub>O<sub>3</sub>N<sub>2</sub> MW, 204

Cryst. from EtOH. M.p. 262°.

Macbeth, Nunan, Traill, *J. Chem. Soc.*, 1926, 1252.

### 5-Phenylbarbituric Acid.

M.p. 258° (250°, 206°).

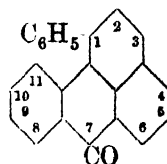
Lund, *Chem. Zentr.*, 1936, **I**, 2096.

Bayer, D.R.P., 249,722, (*Chem. Zentr.*, 1912, **II**, 652).

### *p*-Phenylbenzaldehyde.

See 4-Aldehydodiphenyl.

### 1-Phenylbenzanthrone



C<sub>23</sub>H<sub>14</sub>O

MW, 306

M.p. 186°. Sol. conc.  $\text{H}_2\text{SO}_4$  with orange-red col. showing strong fluor.  $\text{Na}_2\text{Cr}_2\text{O}_7 \rightarrow$  anthraquinone-1-carboxylic acid.

Clar, *Ber.*, 1932, **65**, 858.

I.G., E.P., 286,685, (*Chem. Abstracts*, 1929, **23**, 1139).

Nakanishi, *Chem. Abstracts*, 1934, **28**, 763.

### 2-Phenylbenzanthrone.

Yellow needles from AcOH. M.p. 199–200°. Sol.  $\text{H}_2\text{SO}_4$  to red sol. changing to orange-red.

I.G., E.P., 297,129, (*Chem. Abstracts*, 1929, **23**, 2580).

### 3-Phenylbenzanthrone.

Yellow needles from AcOH. M.p. 182–3° (181°). Ox.  $\rightarrow$  phenyl 1-anthraquinonyl ketone.

Allen, Overbaugh, *J. Am. Chem. Soc.*, 1935, **57**, 1323.

Nakanishi, *Chem. Abstracts*, 1934, **28**, 763.

I.G., D.R.P., 488,607, (*Chem. Abstracts*, 1930, **24**, 2148); E.P., 297,129, (*Chem. Abstracts*, 1929, **23**, 2580).

### 6-Phenylbenzanthrone.

M.p. 186°. Alk.  $\text{KMnO}_4 \rightarrow$  4-phenylantraquinone-1-carboxylic acid.

Allen, Overbaugh, *J. Am. Chem. Soc.*, 1935, **57**, 743.

### 10-Phenylbenzanthrone.

Golden-yellow or yellowish-brown leaflets from  $\text{C}_6\text{H}_6$  or Py.Aq. M.p. 178–9° (170–1°). Very sol.  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ , Py. Mod. sol. AcOH. Spar. sol. EtOH. Very spar. sol. pet. ether. Sol. conc.  $\text{H}_2\text{SO}_4$  with red col. showing golden-yellow fluor.

Scholl, Seer, *Ann.*, 1912, **394**, 150.

Schmidlin, Garcia-Banús, *Ber.*, 1912, **45**, 3184.

### Phenylbenzene.

See Diphenyl.

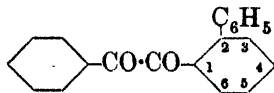
### Phenylbenzhydrylcarbinol.

See 2-Hydroxy-1:1:2-triphenylethane.

### Phenyl benzhydryl Ketone.

See Diphenylbenzoylmethane.

### 2-Phenylbenzil



$\text{C}_{20}\text{H}_{14}\text{O}_2$

MW, 286

Yellow plates from EtOH. M.p. 80°. Ox.  $\rightarrow$  diphenyl-2-carboxylic acid.

Quinoxaline deriv.: m.p. 163°.

Kohler, Nygaard, *J. Am. Chem. Soc.*, 1930, **52**, 4137.

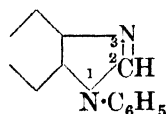
### 4-Phenylbenzil.

Yellow plates from EtOH. M.p. 105°.

Hatt, Pilgrim, Hurran, *J. Chem. Soc.*, 1936, 94.

Gomberg, Natta, *J. Am. Chem. Soc.*, 1929, **51**, 2244.

### 1-Phenylbenziminazole



$\text{C}_{13}\text{H}_{10}\text{N}_2$

MW, 194

Needles from ligroin. M.p. 98°. Sol. MeOH, EtOH. Spar. sol.  $\text{H}_2\text{O}$ .

$B, \text{HCl}, \text{HgCl}_2$ : cryst. from EtOH.Aq. M.p. 145–146.5°.

Fischer, Rigaud, *Ber.*, 1901, **34**, 4204.

Phillips, *J. Chem. Soc.*, 1929, 2823.

### 2-Phenylbenziminazole.

Plates from AcOH, needles from  $\text{H}_2\text{O}$ . M.p. 291° (280°, 285°). Sol. AcOH. Mod. sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Forms metallic derivs. with silver and mercury.

$B, \text{HCl}$ : needles from EtOH. M.p. 303° (above 306°).

Feigl, Gleich, *Monatsh.*, 1928, **49**, 394.

Fischer, Mann-Tiechler, *J. prakt. Chem.*, 1924, **107**, 45.

Guha, Ray, *J. Indian Chem. Soc.*, 1925, **2**, 93.

Hinsberg, Koller, *J. prakt. Chem.*, 1896, **29**, 1498.

### Phenyl benzoate



$\text{C}_{13}\text{H}_{10}\text{O}_2$

MW, 198

Prisms. M.p. 71°. B.p. 314° (299°). Sol. hot EtOH, hot  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ . Heat of comb.  $C_p$  1511.3 Cal.

Rasinski, *J. prakt. Chem.*, 1882, **28**, 62.

Reychler, *Chem. Zentr.*, 1908, **I**, 1042.

Czapek, *Monatsh.*, 1914, **35**, 637.

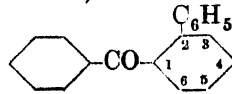
### Phenylbenzoic Acid.

See Diphenyl-carboxylic Acid.

### $\alpha$ -Phenylbenzoin.

See Diphenylbenzoylcarbinol.

**2-Phenylbenzophenone** (2-Benzoyldiphenyl, phenyl o-xenyl ketone)



$\text{C}_{19}\text{H}_{14}\text{O}$

MW, 258



Cryst. from ligroin. M.p. 90°.

Schlenk, Bergmann *et al.*, *Ann.*, 1928, **464**, 34.

**4-Phenylbenzophenone** (4-Benzoyldiphenyl, phenyl p-xenyl ketone).

Cryst. from EtOH. M.p. 102° (106°). B.p. 419–20°/744 mm. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, hot EtOH. CrO<sub>3</sub> → p-benzoylbenzoic acid.

Oxime: needles from EtOH. M.p. 193–4°. Sol. EtOH, Et<sub>2</sub>O.

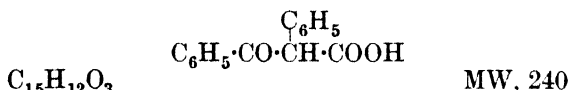
Neitzescu, Isacescu, Ionescu, *Ann.*, 1931, **491**, 217.

Hey, Jackson, *J. Chem. Soc.*, 1936, 805.

Staudinger, Kon, *Ann.*, 1911, **384**, 97.

Wolf, *Ber.*, 1881, **14**, 2032.

**Phenylbenzoylacetic Acid** (2-Keto-1:2-diphenylpropionic acid)



Free acid unknown.

Me ester: C<sub>16</sub>H<sub>14</sub>O<sub>3</sub>. MW, 254. Liq. De-comp. on dist. → stilbene.

Et ester: C<sub>17</sub>H<sub>16</sub>O<sub>3</sub>. MW, 268. Prisms. M.p. 90°. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

1-Menthyl ester: (i) keto form, needles from MeOH. M.p. 116°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold EtOH.  $[\alpha]_D^{20} + 21 \cdot 10^5$  in C<sub>6</sub>H<sub>6</sub>, – 12·1° in EtOH. Et<sub>2</sub>O sol. + FeCl<sub>3</sub> → deep red col. (ii) Enol form:  $[\alpha]_D^{20} - 62 \cdot 83^\circ$  in C<sub>6</sub>H<sub>6</sub>, – 62·6° in EtOH.

Amide: C<sub>15</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 239. Needles from EtOH. M.p. 178°. Sol. EtOH. Spar. sol. Et<sub>2</sub>O, hot H<sub>2</sub>O.

Nitrile: C<sub>15</sub>H<sub>11</sub>ON. MW, 221. Plates from AcOH. M.p. 93–4°. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, hot pet. ether. Alk. hyd. → benzoic acid + benzyl cyanide. Conc. H<sub>2</sub>SO<sub>4</sub> at 120° → amide. Conc. HCl at 150° → deoxybenzoin. Boiling dil. acids → benzoic + phenylacetic acids.

Anilide: needles from EtOH. M.p. 168–9°.

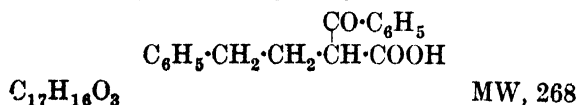
Oxime: cryst. M.p. 138–9°.

Rupe, Gisiger, *Helv. Chim. Acta*, 1925, **8**, 349.

Ghosh, *J. Chem. Soc.*, 1916, **109**, 117.

Walther, Schickler, *J. prakt. Chem.*, 1897, **55**, 314.

**3-Phenyl-1-benzoylbutyric Acid**

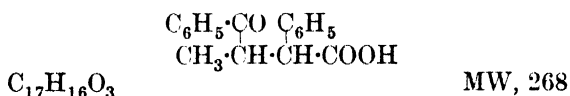


Free acid not isolated.

Et ester: C<sub>19</sub>H<sub>20</sub>O<sub>3</sub>. MW, 296. B.p. 225–30°/11 mm.

Auwers, Möller, *J. prakt. Chem.*, 1925, **109**, 150.

**1-Phenyl-2-benzoylbutyric Acid**

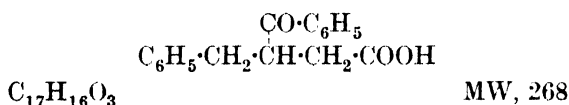


Exists in two forms. (i) Needles from CHCl<sub>3</sub>-ligroin. M.p. 145°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. (ii) Needles from CHCl<sub>3</sub>-ligroin. M.p. 131°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. ligroin, cold H<sub>2</sub>O.

Me ester: C<sub>18</sub>H<sub>18</sub>O<sub>3</sub>. MW, 282. Exists in two forms. (i) Plates from MeOH. M.p. 105°. (ii) M.p. 87°.

Reimer, Reynolds, *Am. Chem. J.*, 1912, **48**, 216, 218.

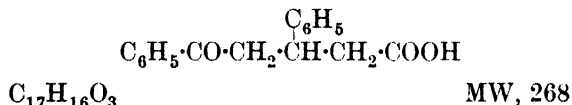
**3-Phenyl-2-benzoylbutyric Acid**



Needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 100–1°.

Borsche, *Ber.*, 1914, **47**, 1114.

**2-Phenyl-3-benzoylbutyric Acid** (β-Phenacylhydrocinnamic acid)



Needles or plates from H<sub>2</sub>O, prisms from AcOH. M.p. 160° (152–153·5°). Sol. EtOH, AcOH. Spar. sol. Et<sub>2</sub>O. Hot Ac<sub>2</sub>O → lactone. Me ester: C<sub>18</sub>H<sub>18</sub>O<sub>3</sub>. MW, 282. Needles. M.p. 94°.

Et ester: C<sub>19</sub>H<sub>20</sub>O<sub>3</sub>. MW, 296. M.p. 59–61°.

Lactone: C<sub>17</sub>H<sub>14</sub>O<sub>2</sub>. MW, 250. Needles. Sol. Et<sub>2</sub>O. Hot alc. NaOH regenerates the acid.

Oxime: m.p. 144–145·5°.

Semicarbazone: cryst. from 90% EtOH. M.p. 220° (212·5–213°).

Stobbe, *Ber.*, 1901, **34**, 655.

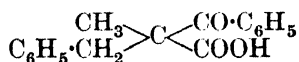
Vorländer, Knötzsch, *Ann.*, 1897, **294**, 332.

Kohler, Steele, *J. Am. Chem. Soc.*, 1919, **41**, 1103.

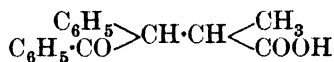
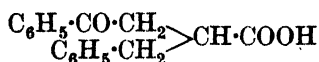
Qudrat-i-Khuda, *J. Indian Chem. Soc.*, 1931, **8**, 220.

**3-Phenyl-3-benzoylbutyric Acid** (*2-Desylpropionic acid*)C<sub>17</sub>H<sub>16</sub>O<sub>3</sub> MW, 268Needles from EtOH. M.p. 136° (133–4°).  
Sol. EtOH, AcOH. Insol. H<sub>2</sub>O.*Me ester*: needles. M.p. 63–4°.*Et ester*: needles. M.p. 33–4°.Knoevenagel, *Ber.*, 1888, **21**, 1351.Meerwein, *J. prakt. Chem.*, 1918, **97**, 261.**Phenylbenzoyldiazomethane.**

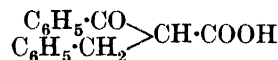
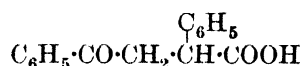
See Azibenzil.

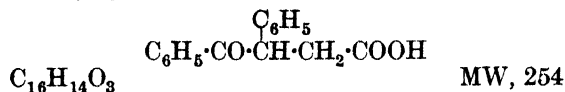
**2-Phenyl-1-benzoylisobutyric Acid**  
(*Methylbenzylbenzoylacetic acid*, 1-benzyl-1-benzoylpropionic acid)C<sub>17</sub>H<sub>16</sub>O<sub>3</sub> MW, 268

Free acid not isolated.

*Et ester*: C<sub>19</sub>H<sub>20</sub>O<sub>3</sub>. MW, 296. Oil. B.p. 223–8°/19 mm.Hope, Perkin, *J. Chem. Soc.*, 1909, **95**, 2046.**2-Phenyl-2-benzoylisobutyric Acid** (*1-Desylpropionic acid*)C<sub>17</sub>H<sub>16</sub>O<sub>3</sub> MW, 268Needles from EtOH. M.p. 220° (213–5°).  
Insol. H<sub>2</sub>O.Knoevenagel, *Ber.*, 1888, **21**, 1353.Japp, Michie, *J. Chem. Soc.*, 1903, **83**, 299.**2-Phenyl-2'-benzoylisobutyric Acid** (*α-Phenacylhydrocinnamic acid*, 1-benzyl-2-benzoylpropionic acid)C<sub>17</sub>H<sub>16</sub>O<sub>3</sub> MW, 268Needles from MeOH. M.p. 176° (169–5°).  
Sol. EtOH, CHCl<sub>3</sub>, AcOH. Mod. sol. MeOH.  
Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O, pet. ether.  
Sublimes.*Me ester*: C<sub>18</sub>H<sub>18</sub>O<sub>3</sub>. MW, 282. Prisms from MeOH. M.p. 68–5°.Thiele, Mayr, *Ann.*, 1899, **306**, 186.Klobb, *Bull. soc. chim.*, 1897, **17**, 411.Kohler, *Ber.*, 1905, **38**, 1206.

Dict. of Org. Comp.—III.

**2-Phenyl-1-benzoylpropionic Acid** (*Benzylbenzoylacetic acid*, *α-benzoylhydrocinnamic acid*)C<sub>16</sub>H<sub>14</sub>O<sub>3</sub> MW, 254*Me ester*: C<sub>17</sub>H<sub>16</sub>O<sub>3</sub>. MW, 268. B.p. 250–55°/50 mm.*Et ester*: C<sub>18</sub>H<sub>18</sub>O<sub>3</sub>. MW, 282. B.p. 265–70°/80 mm., 218–20°/12 mm.*l-Menthyl ester*: needles. M.p. 117°. [α]<sub>D</sub><sup>20</sup> – 60–83° in C<sub>6</sub>H<sub>6</sub>.Perkin, Calman, *J. Chem. Soc.*, 1886, **49**, 155.Perkin, Stenhouse, *J. Chem. Soc.*, 1891, **59**, 1006.Jacobson, Ghosh, *J. Chem. Soc.*, 1915, **107**, 961.**1-Phenyl-2-benzoylpropionic Acid**  
(*Phenylphenacylacetic acid*)C<sub>16</sub>H<sub>14</sub>O<sub>3</sub> MW, 254*d.*Plates from Et<sub>2</sub>O. M.p. 176–8°. [α]<sub>D</sub> + 157.3° in AcOEt.*Semicarbazone*: needles from MeOH. M.p. 107–10°, resolidifying on further heating, melting again above 160°.*l.*Plates from Et<sub>2</sub>O. M.p. 176–8°. [α]<sub>D</sub><sup>18</sup> – 157.6° in AcOEt.*dl.*Prisms from EtOH. M.p. 153° (160°). Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. hot H<sub>2</sub>O.  
*Me ester*: cryst. from MeOH. M.p. 104°.*Et ester*: needles. M.p. 37–8°.*Amide*: C<sub>16</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 253. Needles from EtOH. M.p. 149°.*Nitrile*: C<sub>16</sub>H<sub>13</sub>ON. MW, 235. Plates from EtOH. M.p. 127–5°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.*Oxime*: plates + 1 mol. C<sub>6</sub>H<sub>6</sub> from C<sub>6</sub>H<sub>6</sub>. M.p. 83–7°.*Semicarbazone*: cryst. powder from EtOH. M.p. 189–91°.Lapworth, Wechsler, *J. Chem. Soc.*, 1910, **97**, 41.Allen, Kimball, *Organic Syntheses*, 1930, **X**, 80.Hann, Lapworth, *J. Chem. Soc.*, 1904, **85**, 1368.Rupe, Schneider, *Ber.*, 1895, **28**, 962.

**2-Phenyl-2-benzoylpropionic Acid** ( $\beta$ -Benzoylhydrocinnamic acid, desylacetic acid)

Cryst. from EtOH. M.p. 162°. Fuming HI  $\rightarrow$  2:3-diphenylbutyric acid. NaHg  $\rightarrow$  2:3-diphenyl-3-butyrolactone.

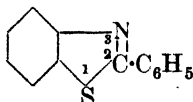
Me ester:  $\text{C}_{17}\text{H}_{16}\text{O}_3$ . MW, 268. Cryst. from MeOH. M.p. 49°.

Thiele, Straus, *Ann.*, 1901, **319**, 164.

Anschütz, Walter, *Ann.*, 1907, **354**, 147.

**2-Phenyl- $\gamma$ -benzpyrone.**

See Flavone.

**2-Phenylbenzthiazole**

Needles from EtOH.Aq. M.p. 114°.

Methiodide: m.p. 218°.

Methopicate: orange-yellow plates. M.p. 125-6°.

Methoperchlorate: needles. M.p. 220°.

Methochloroplatinate: m.p. 243° decomp.

Dibromide: m.p. 119°.

Tetrabromide: m.p. 134-6°.

Hexabromide: m.p. 156° decomp.

$\text{C}_{13}\text{H}_9\text{NS} \cdot \text{C}_6\text{H}_5(\text{NO}_2)_3 \cdot 1:3:5$ : yellow needles. M.p. 106°.

Chandri, Desai, Hunter, *J. Indian Chem. Soc.*, 1934, **11**, 252.

Hunter, *J. Chem. Soc.*, 1930, 138.

Clark, *J. Chem. Soc.*, 1928, 2316.

Chapman, *ibid.*, 1894.

Bogert, Abrahamson, *J. Am. Chem. Soc.*, 1922, **44**, 831.

**Phenylbenzylacetic Acid.**

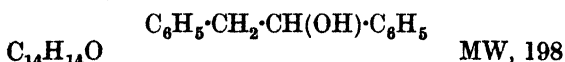
See 1:2-Diphenylpropionic Acid.

**Phenylbenzyl Alcohol.**

See Hydroxymethyl-diphenyl.

**Phenylbenzylamine.**

See Benzylaniline.

**Phenylbenzylcarbinol** ( $\alpha$ -Hydroxydibenzyl, 1-hydroxy-1:2-diphenylethane)

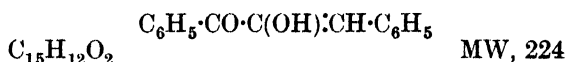
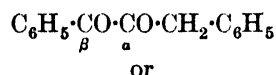
Needles from EtOH.Aq. or AcOH. M.p. 67-8° (62°). B.p. 167-70°/10 mm. Dist. at ord. press.  $\rightarrow$  stilbene.  $\text{HNO}_3$  (D 1:3)  $\rightarrow$  deoxybenzoin. H (+ Ni) at 360°  $\rightarrow$  dibenzyl.

Et ether:  $\alpha$ -ethoxydibenzyl.  $\text{C}_{16}\text{H}_{18}\text{O}$ . MW, 226. B.p. 149-51°/10 mm. Hot dil.  $\text{H}_2\text{SO}_4$   $\rightarrow$  stilbene.

Schmidlin, Garcia-Banús, *Ber.*, 1912, **45**, 3199.

Marshall, *J. Chem. Soc.*, 1915, **107**, 520.

Limpricht, Schwanert, *Ann.*, 1870, **155**, 62.

**Phenyl benzyl Diketone** (Benzoylphenacetyl, phenylbenzylglyoxal, 1:3-diphenylpropandione-1:2, 1:2-diketo-1:3-diphenylpropane)

Three modifications have been described:

(i) Yellow needles. M.p. 67°.

(ii) Yellowish-white cryst. M.p. 90°.

(iii) Lemon-yellow needles. M.p. 35-6°. B.p. 191-2°/15 mm.

(i) and (ii) are probably stereoisomers of the enolic form. (iii) is the ketonic form.

Readily oxidised in air.

$\alpha$ -Oxime:  $\omega$ -isonitroso- $\omega$ -benzylacetophenone. Plates from  $\text{C}_6\text{H}_6$ . M.p. 126°. Sol. alkalis with yellow col.

Dioxime: phenylbenzylglyoxime. Plates from AcOH. M.p. 207°.

Di-phenylhydrazone: m.p. 131°.

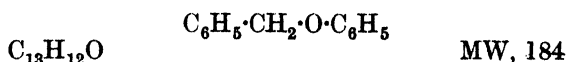
Wieland, *Ber.*, 1903, **36**, 3018.

Widman, *Ber.*, 1916, **49**, 484.

Jorländer, *Ber.*, 1917, **50**, 416.

Moureu, *Ann. chim.*, 1930, **14**, 301.

Kohler, Barnes, *J. Am. Chem. Soc.*, 1934, **56**, 211.

**Phenyl benzyl Ether**

Leaflets from EtOH. M.p. 40°. B.p. 286-7°, 178-9°/35 mm., 124-5°/4 mm. Conc. HCl at 100°  $\rightarrow$  phenol + benzyl chloride.

van Duzee, Adkins, *J. Am. Chem. Soc.*, 1935, **57**, 148.

Baw, *J. Indian Chem. Soc.*, 1926, **3**, 103.

Powell, Adams, *J. Am. Chem. Soc.*, 1920, **42**, 656.

**sym.-Phenylbenzylethylene.**

See 1:3-Diphenylpropylene.

**Phenylbenzylglycollic Acid.**

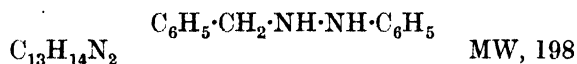
See 1:2-Diphenyl-lactic Acid.

**Phenylbenzylglyoxal.**

See Phenyl benzyl Diketone.

**Phenylbenzylglyoxime.**

See under Phenyl benzyl Diketone.

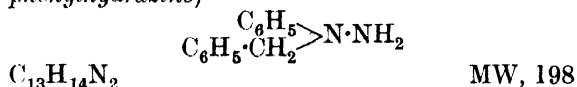
**sym.-Phenylbenzylhydrazine** ( $\beta$ -Benzylphenylhydrazine)

Colourless cryst. M.p. 35°. B.p. about 290° decomp. Very sol. prac. all org. solvents. Oxidises in air to benzaldehyde phenylhydrazone.

*B.HCl*: leaflets from  $\text{H}_2\text{O}$ . M.p. 205° (193–5°). Spar. sol. EtOH, cold  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ . *B.(COOH)\_2*: m.p. 190°.

Ponzio, Valente, *Gazz. chim. ital.*, 1908, 38, i, 525.

Schlenk, *J. prakt. Chem.*, 1908, 78, 51.

**unsym.-Phenylbenzylhydrazine** ( $\alpha$ -Benzylphenylhydrazine)

Colourless oil. B.p. 216–18°/38 mm., 207–8°/10 mm. Part. decomp. on standing.

*B.HCl*: needles from  $\text{H}_2\text{O}$ . M.p. 170° (166–7°).

*d-Camphor-β-sulphonic acid salt*: prisms from  $\text{AcOEt-Et}_2\text{O}$ . M.p. 156–7°.

*Methochloride*: prisms. M.p. 158–9° decomp. Very sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . *B.AuCl\_3*: yellow prisms from dil. HCl. M.p. 134° decomp. *B\_2.PtCl\_4*: orange needles from dil. HCl. M.p. 162° decomp.

*Methiodide*: cryst. from EtOH. M.p. 122° decomp. *B\_2.HgI\_2*: prisms from MeOH. M.p. 135–6° decomp. *B.HgI\_2*: yellow prisms from EtOH. M.p. 128–9° decomp.

Ofner, *Monatsh.*, 1904, 25, 599.

Milrath, *Monatsh.*, 1908, 29, 910 (Footnote 2).

Ruff, Ollendorff, *Ber.*, 1899, 32, 3235 (Footnote 2).

Ponzio, Valente, *Gazz. chim. ital.*, 1908, 38, i, 520.

Michaelis, Philips, *Ann.*, 1889, 252, 286.

Singh, *J. Chem. Soc.*, 1914, 105, 1976.

**Phenylbenzylideneacetone.**

See Acetostilbene and Benzyl styryl Ketone.

**Phenylbenzylidenelactic Acid.**

See 1-Hydroxy-2:3-diphenylvinylacetic Acid.

**Phenyl benzyl Ketone.**

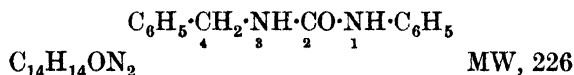
See Deoxybenzoin.

**Phenylbenzylnitrosamine.**

See under Benzylaniline.

**Phenyl benzyl sulphide.**

See under Benzyl Mercaptan.

**sym.-Phenylbenzylurea**

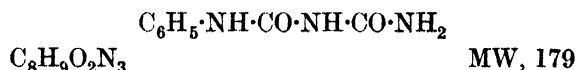
Needles from EtOH. M.p. 170° (168°). Sol. EtOH. Insol.  $\text{H}_2\text{O}$ .

1-N-Me:  $\text{C}_{15}\text{H}_{16}\text{ON}_2$ . MW, 240. M.p. 84°. Sol. EtOH, AcOH,  $\text{C}_6\text{H}_6$ .

Ley, Krafft, *Ber.*, 1907, 40, 703.

Thiele, Pickard, *Ann.*, 1899, 309, 203.

Kühn, Riesenfeld, *Ber.*, 1891, 24, 3817.

**ω-Phenylbiuret** (*Allophanic acid anilide, 1-phenylbiuret, allophanylaniline*)

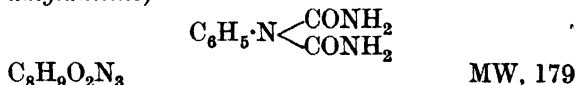
Cryst. from EtOH.Aq. M.p. 167° (various m.p.'s between 156–65° are recorded, due to decomp. on melting). Sol. EtOH. Insol.  $\text{Et}_2\text{O}$ . Does not give biuret reaction. Heat  $\rightarrow \text{NH}_3$  + cyanuric acid + carbanilide. Heat with aniline  $\rightarrow$  carbanilide.

Davis, Blanchard, *J. Am. Chem. Soc.*, 1929, 51, 1809.

Dains, Wertheim, *J. Am. Chem. Soc.*, 1920, 42, 2307.

Gatewood, *J. Am. Chem. Soc.*, 1925, 47, 410.

Blair, *J. Am. Chem. Soc.*, 1934, 56, 904.

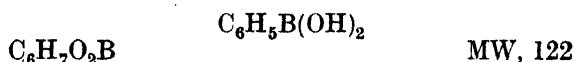
**ms.-Phenylbiuret** (*3-Phenylbiuret, dicarb-amylniline*)

Needles or prisms from hot  $\text{H}_2\text{O}$ . M.p. 192°. Very sol. EtOH, boiling  $\text{H}_2\text{O}$ . Heat with aniline  $\rightarrow$  carbanilide.  $\text{KOH}$  + a little  $\text{CuSO}_4$   $\rightarrow$  reddish-violet col.

See last reference above and also

Schiff, *Ann.*, 1907, 352, 78.

Weith, *Ber.*, 1877, 10, 1744.

**Phenylboric Acid**

Needles from  $\text{H}_2\text{O}$ . M.p. 221° (216°). Sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Volatile in steam.

Chlorine water  $\rightarrow$  chlorobenzene.  $\text{H}_2\text{O}_2 \rightarrow$  phenol. Boiling conc.  $\text{HCl} \rightarrow$  benzene.

Ainley, Challenger, *J. Chem. Soc.*, 1930, 2171.

Seaman, Johnson, *J. Am. Chem. Soc.*, 1931, 53, 713.

Bean, Johnson, *J. Am. Chem. Soc.*, 1932, 54, 4417.

Krause, Nitsche, *Ber.*, 1922, 55, 1264.

Krause, D.R.P., 371,467, (*Chem. Abstracts*, 1924, 18, 992).

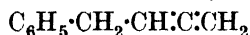
### Phenylbromoacetic Acid.

See  $\alpha$ -Bromophenylacetic Acid.

### Phenyl $\omega$ -bromobenzyl Ketone.

See Desyl bromide.

### 4-Phenyl-1 : 3-butadiene (1-Benzylallene)



$\text{C}_{10}\text{H}_{10}$  MW, 130

B.p.  $72-3^\circ/17$  mm.  $D_4^{20}$  0.9220.  $n_D^{20}$  1.5460.

Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, 55, 2816.

### 1-Phenyl-1 : 3-butadiene (1-Phenylerythrene)



$\text{C}_{10}\text{H}_{10}$  MW, 130

*Cis* :

B.p.  $86^\circ/11$  mm.  $n_D^{28}$  1.5950.

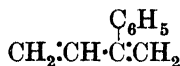
*Trans* :

B.p.  $76^\circ/11$  mm.  $n_D^{28}$  1.5920.

Muskat, Herrmann, *J. Am. Chem. Soc.*, 1931, 53, 266.

Bergmann, Bondi, *Ber.*, 1931, 64, 1479.

### 2-Phenyl-1 : 3-butadiene (2-Phenylerythrene, phenoprene)



$\text{C}_{10}\text{H}_{10}$  MW, 130

B.p.  $60-1^\circ/17$  mm.  $D_4^{20}$  0.9226.  $n_D^{20}$  1.5489.

*Dimer* : needles from  $\text{MeOH}$ . M.p.  $62^\circ$ . B.p.  $220-5^\circ/10$  mm.

Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, 55, 2816.

### 4-Phenyl-1 : 3-butadiene-1-carboxylic Acid.

See 2-Styrylacrylic Acid.

### 4-Phenyl-1 : 3-butadiene-1 : 1-dicarboxylic Acid.

See Cinnamylidenemalonic Acid.

### Phenylbutandiol.

See Phenylbutylene Glycol and Phenyltetramethylene Glycol.

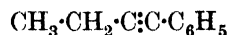
### Phenylbutane.

See Butylbenzene.

### Phenylbutenol.

See Methylstyrylcarbinol, 2-Phenylallylcarbinol, Propenylphenylcarbinol, and Allylphenylcarbinol.

### 1-Phenylbutine-1 (1-Ethyl-2-phenylacetylene)



$\text{C}_{10}\text{H}_{10}$  MW, 130

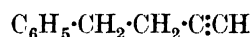
B.p.  $201-3^\circ$ ,  $87-90^\circ/18$  mm.  $D^{18}$  0.915.  $n_D^{18}$  1.537.

Truchet, *Ann. chim.*, 1931, 16, 397.

Morgan, *J. Chem. Soc.*, 1876, 29, 162.

Bergmann, Bondi, *Ber.*, 1933, 66, 286.

### 4-Phenylbutine-1 ( $\beta$ -Phenethylacetylene)



$\text{C}_{10}\text{H}_{10}$  MW, 130

B.p.  $189-91^\circ/758$  mm.,  $83^\circ/15$  mm.  $D_4^{20}$  0.928,  $D_4^{18}$  0.918.  $n_D^{20}$  1.5212. Heat of comb.  $\text{C}_v$  1340-1 Cal.

André, *Bull. soc. chim.*, 1911, 9, 193.

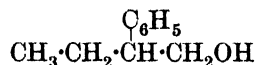
Moureu, André, *Ann. chim.*, 1914, 1, 116.

Bert, Dorier, Lamy, *Compt. rend.*, 1925, 181, 555.

### 1-Phenyl-*n*-butyl Alcohol.

See Propylphenylcarbinol.

### 2-Phenyl-*n*-butyl Alcohol ( $\alpha$ -Hydroxymethylpropylbenzene)



$\text{C}_{10}\text{H}_{14}\text{O}$  MW, 150

*dl.*

B.p.  $124-7^\circ/25$  mm.,  $120-1^\circ/15$  mm.  $D_4^{18}$  0.989.  $n_D^{18}$  1.43.

*Benzoyl* : m.p.  $115-16^\circ$ . B.p.  $202-3^\circ/18$  mm.

*Phenylurethane* : m.p.  $58-9^\circ$ .

*d.*

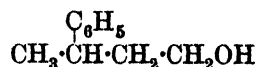
$[\alpha]_D^{25} + 1.8^\circ$ ,  $[\alpha]_D^{30} + 2.54^\circ$  ( $+ 1.11^\circ$ ).

Lagerrev, *Chem. Zentr.*, 1935, II, 2809.

Levene, Marker, *J. Biol. Chem.*, 1935, 108, 415.

Rampart, Amagat, *Ann. chim.*, 1927, 8, 284.

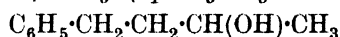
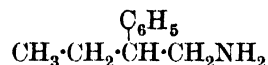
### 3-Phenyl-*n*-butyl Alcohol



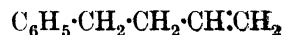
$\text{C}_{10}\text{H}_{14}\text{O}$  MW, 150

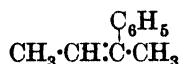
*l.*

B.p.  $117^\circ/8$  mm.  $D_4^{25}$  0.986.  $n_D^{25}$  1.5201.  $[\alpha]_D^{25} - 9.15^\circ$  ( $[\alpha]_D^{20} - 2.89^\circ$ ).

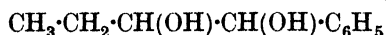
*dl.*B.p. 138–40°/33 mm., 125.5–128°/13 mm., 110–11°/6 mm.  $D_D^{20}$  0.9834.Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 762; 1935, **108**, 413.Wojcik, Adkins, *J. Am. Chem. Soc.*, 1933, **55**, 4943.Rupe, van Walraven, *Helv. Chim. Acta*, 1930, **13**, 369.**4-Phenyl-*n*-butyl Alcohol** ( $\delta$ -Hydroxybutylbenzene) $C_{10}H_{14}O$  MW, 150Oil. B.p. 140°/14 mm. Insol.  $H_2O$ .  $D_4^{20}$  1.001.*Me ether*:  $C_{11}H_{16}O$ . MW, 164. B.p. 108°/11 mm.*Nitrite*:  $C_6H_5 \cdot [CH_2]_4 \cdot O \cdot NO$ . B.p. 125–30°/15 mm.*p*-Nitrobenzoyl: m.p. 18–20°.*Phenylurethane*: m.p. 51–2°.Kirner, *J. Am. Chem. Soc.*, 1926, **48**, 1112.v. Braun, *Ber.*, 1911, **44**, 2871.v. Braun, *Deutsch, Ber.*, 1912, **45**, 2176.**2-Phenyl-*sec.*-*n*-butyl Alcohol.***See* Methyl ethyl phenylcarbinol.**4-Phenyl-*sec.*-*n*-butyl Alcohol** ( $\gamma$ -Hydroxybutylbenzene, methyl- $\beta$ -phenylethylcarbinol) $C_{10}H_{14}O$  MW, 150*d.*B.p. 132°/14 mm.  $D_4^{20}$  0.9788.  $n_D^{20}$  1.5168. Volatile in steam.*Formyl*: b.p. 120°/15 mm.  $D_4^{20}$  1.0083.*Acetyl*: b.p. 130°/15 mm.  $D_4^{20}$  0.9854.*Propionyl*: b.p. 141°/16 mm.  $D_4^{20}$  0.9790.*l.*B.p. 132°/14 mm.  $n_D^{20}$  1.5168.  $[\alpha]_D^{20}$  – 19.45° in EtOH. Volatile in steam.*dl.*Oil with aromatic odour. B.p. 238°/750 mm., 123–4°/15 mm.  $D_4^{20}$  0.9899.  $n_D^{20}$  1.517.*Acetyl*: b.p. 123–4°/15 mm.  $D_4^{18}$  0.991.  $n_D^{18}$  1.4895.*Phenylurethane*: m.p. 116–17° (113°).Hewitt, Kenyon, *J. Chem. Soc.*, 1925, **127**, 1094.Zechmeister, Rom, *Ann.*, 1929, **468**, 126.Schlenk, Bergmann, *Ann.*, 1930, **479**, 73.Packendorff, *Ber.*, 1934, **67**, 906.Pickard, Kenyon, *J. Chem. Soc.*, 1914, **105**, 1125.**Phenyl-*tert.*-butyl Alcohol.***See* Dimethylbenzylcarbinol.**1- and 4-Phenylbutylamines.***See* Aminobutylbenzene.**2-Phenyl-*n*-butylamine** $C_{10}H_{15}N$  MW, 149

B.p. 110°/13 mm.

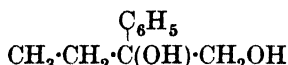
*B, HCl*: cryst. from Et<sub>2</sub>O. M.p. 156°.Rampart, Amagat, *Ann. chim.*, 1927, **8**, 285.**1-Phenyl-1-butylene** ( $\alpha$ -Butenylbenzene) $C_{10}H_{12}$  MW, 132B.p. 190–3° (186–7°), 89–90°/15 mm., 78°/12 mm., 70–1°/8 mm.  $D_4^{20}$  0.9106.  $n_D^{18}$  1.5381.Na + EtOH  $\rightarrow$  butylbenzene.*Nitrosite*: m.p. 124°.*Dibromide*: m.p. 70°.Auwers, Roth, Eisenlohr, *Ann.*, 1910, **373**, 282.Klages, *Ber.*, 1904, **37**, 2312.Lévy, Dvoretzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1769.For *cis*- and *trans*- forms seeMuskat, Knapp, *Ber.*, 1931, **64**, 785.**4-Phenyl-1-butylene** ( $\gamma$ -Butenylbenzene) $C_{10}H_{12}$  MW, 132B.p. 177–8°/754 mm., 64°/10 mm.  $D_4^{20}$  0.8831.  $n_D^{20}$  1.5059. Heat of comb.  $C_6$  1356.8 Cal.  $KMnO_4$  in  $Me_2CO \rightarrow$  hydrocinnamic acid.André, *Bull. soc. chim.*, 1911, **9**, 193.Riiber, *Ber.*, 1911, **44**, 2392.Gilman, McGlumphy, *Bull. soc. chim.*, 1928, **43**, 1326.**1-Phenyl-2-butylene** ( $\beta$ -Butenylbenzene, crotonylbenzene) $C_{10}H_{12}$  MW, 132Oily liq. B.p. 176°, 81–2°/21 mm., 70°/12 mm.  $D_4^{20}$  0.8831.  $n_D^{20}$  1.5101. Alc. KOH  $\rightarrow$  1-phenyl-1-butylene. Ozone in presence of  $H_2O \rightarrow$  phenylacetaldehyde.Auwers, Roth, Eisenlohr, *Ann.*, 1911, **385**, 108.Klages, *Ber.*, 1904, **37**, 2310.Straus, *Ann.*, 1905, **342**, 257.

**2-Phenyl-2-butylene** ( $\alpha$ -Methylpropenylbenzene) $\text{C}_{10}\text{H}_{12}$  MW, 132

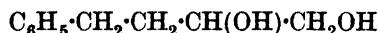
B.p. 188–9°, 80–1°/20 mm.  $D_4^{20}$  0.9088.  $n_D^{19}$  1.5339. Heat of comb.  $\text{C}_6$  1352.8 Cal.  $\text{KMnO}_4$  in  $\text{H}_2\text{SO}_4 \rightarrow$  acetophenone.

Klages, *Ber.*, 1902, **35**, 2641, 3508.Haller, Bauer, *Ann. chim.*, 1918, **9**, 12.Thorpe, Wood, *J. Chem. Soc.*, 1913, **103**, 1578.**1-Phenyl- $\alpha$ -butylene Glycol** (1-Phenyl-1 : 2-butandiol,  $\alpha\beta$ -dihydroxybutylbenzene) $\text{C}_{10}\text{H}_{14}\text{O}_2$  MW, 166

Two forms. (i) M.p. 40–1°. (ii) B.p. 205–8°/72 mm.

Lévy, Dvoretzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1774.**2-Phenyl- $\alpha$ -butylene Glycol** (2-Phenyl-1 : 2-butandiol) $\text{C}_{10}\text{H}_{14}\text{O}_2$  MW, 166

Cryst. from ligroin. M.p. 56°. B.p. 165°/23 mm. Sol.  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O. At 250–300°  $\rightarrow$  diphenylacetaldehyde. At 400–50°  $\rightarrow$  deoxybenzoin.

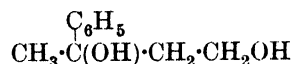
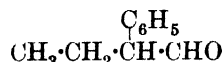
Ramart-Lucas, Salmon-Legagneur, *Bull. soc. chim.*, 1932, **51**, 1078.Stoermer et al., *Ber.*, 1906, **38**, 2300.**4-Phenyl- $\alpha$ -butylene Glycol** (4-Phenyl-1 : 2-butandiol,  $\gamma\delta$ -dihydroxybutylbenzene) $\text{C}_{10}\text{H}_{14}\text{O}_2$  MW, 166

Viscous oil. B.p. 180°/13 mm.

v. Braun, *Ber.*, 1923, **56**, 2182.**1-Phenyl- $\beta$ -butylene Glycol** (1-Phenyl-1 : 3-butandiol,  $\alpha\gamma$ -dihydroxybutylbenzene) $\text{C}_{10}\text{H}_{14}\text{O}_2$  MW, 166

Powder. Sinters at 60°. M.p. about 73.5°. B.p. 175–8°/21 mm., 168–9°/13 mm., 129–31°/2 mm. Sol. EtOH,  $\text{C}_6\text{H}_6$ , ligroin, hot Et<sub>2</sub>O. Spar. sol.  $\text{H}_2\text{O}$ .

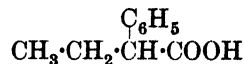
*Diacyl*: b.p. 157°/10 mm., 140°/2 mm. Decomp. on dist. at ord. press.

Michael, Ross, *J. Am. Chem. Soc.*, 1931, **53**, 2412.Sprague, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 2670.Bauer, *Compt. rend.*, 1912, **154**, 1093.**3-Phenyl- $\beta$ -butylene Glycol** (3-Phenyl-1 : 3-butandiol) $\text{C}_{10}\text{H}_{14}\text{O}_2$  MW, 166B.p. 130°/1 mm.  $D^{20}$  1.0865.  $n_D^{20}$  1.5341.St. Pfau, Plattner, *Helv. Chim. Acta*, 1932, **15**, 1266.**1-Phenylbutyraldehyde** (Ethylphenylacetaldehyde) $\text{C}_{10}\text{H}_{12}\text{O}$  MW, 148

Oil. B.p. 211°, 104–6°/15 mm.

*Semicarbazone*: m.p. 155°.Stoermer, *Ber.*, 1906, **39**, 2300.**2-Phenylbutyraldehyde.**See  $\beta$ -Methylhydrocinnamaldehyde.**3-Phenylbutyraldehyde** $\text{C}_{10}\text{H}_{12}\text{O}$  MW, 148

B.p. 129–31°/17 mm. (120–2°/16 mm.).

*Semicarbazone*: leaflets from MeOH. M.p. 104–5°.*Di-Me acetal*: b.p. 121–4°/9 mm.v. Braun, Anton, Fischer, Keller, Manz, *Ber.*, 1934, **67**, 225.v. Braun, Kruber, *Ber.*, 1912, **45**, 394.**1-Phenylbutyric Acid** (Ethylphenylacetic acid) $\text{C}_{10}\text{H}_{12}\text{O}_2$  MW, 164Plates from Et<sub>2</sub>O. M.p. 42°. B.p. 270°.*Me ester*:  $\text{C}_{11}\text{H}_{14}\text{O}_2$ . MW, 178. B.p. 228°.*Amide*:  $\text{C}_{10}\text{H}_{13}\text{ON}$ . MW, 163. M.p. 86°.

*Nitrile*:  $\text{C}_{10}\text{H}_{11}\text{N}$ . MW, 145. B.p. 238–40°, 114–15°/15 mm.  $D^{14}$  0.977. Sol. EtOH,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . Volatile in steam.

Rüber, *Ber.*, 1903, **36**, 1406.Rupe, *Ann.*, 1909, **369**, 334.Neure, *Ann.*, 1889, **250**, 154.Bodroux, Taboury, *Bull. soc. chim.*, 1910, **7**, 667.

**2-Phenylbutyric Acid.**See  $\beta$ -Methylhydrocinnamic Acid.**3-Phenylbutyric Acid** (2-Benzylpropionic acid)
$$\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{COOH}$$

$$\text{C}_{10}\text{H}_{12}\text{O}_2 \quad \text{MW, 164}$$

Leaflets from  $\text{H}_2\text{O}$ . M.p.  $52^\circ$ . B.p.  $290^\circ$ ,  $171^\circ/15$  mm. Sol. EtOH,  $\text{Et}_2\text{O}$ . Mod. sol. warm  $\text{H}_2\text{O}$ .

Et ester:  $\text{C}_{12}\text{H}_{16}\text{O}_2$ . MW, 192. B.p.  $130-1^\circ/10$  mm.  $D_4^{20}$  1.001.

l-Menthyl ester: b.p.  $205^\circ/10$  mm.  $[\alpha]_D^{20}$   $-57.0^\circ$  in  $\text{C}_6\text{H}_6$ .

Chloride:  $\text{C}_{10}\text{H}_{11}\text{OCl}$ . MW, 182.5. B.p.  $140-2^\circ/12$  mm.,  $119^\circ/9$  mm.

Amide:  $\text{C}_{10}\text{H}_{13}\text{ON}$ . MW, 163. Plates from  $\text{H}_2\text{O}$ . M.p.  $84.5^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ .

Nitrile:  $\text{C}_{10}\text{H}_{11}\text{N}$ . MW, 145. B.p.  $142-5^\circ/16$  mm.,  $129-31^\circ/10$  mm.

Overbaugh, Allen, Martin, Fieser, *Organic Syntheses*, 1935, XV, 64.

Richard, *Compt. rend.*, 1935, 200, 1945.

Amagat, *Bull. soc. chim.*, 1927, 41, 942.

Auwers, Möller; *J. prakt. Chem.*, 1925, 109, 138.

v. Braun, *Ber.*, 1910, 43, 2844; 1911, 44, 2871.

Rupe, *Ann.*, 1913, 395, 118.

**Phenylbutyrolactam.**

See Phenylpyrrolidone.

**3-Phenylbutyrolactone.**

See under 3-Hydroxy-3-phenylbutyric Acid.

**3-Phenyl- $\gamma$ -butyrolactone-2-carboxylic Acid.**See  $\gamma$ -Phenylparaconic Acid.**Phenylcacodyl** (Tetraphenyldiarsine)
$$(\text{C}_6\text{H}_5)_2\text{As}\cdot\text{As}(\text{C}_6\text{H}_5)_2$$

$$\text{C}_{24}\text{H}_{20}\text{As}_2 \quad \text{MW, 458}$$

Cryst. M.p.  $135^\circ$ . B.p.  $200^\circ/1$  mm. Sol. EtOH. Less sol.  $\text{Et}_2\text{O}$ . Sol. 100 parts  $\text{C}_6\text{H}_6$  at  $20^\circ$ .

Michaelis, *Ann.*, 1902, 321, 148.

Michaelis, Schulte, *Ber.*, 1882, 15, 1954.

Porter, Borgstrom, *J. Am. Chem. Soc.*, 1919, 41, 2049.

**5-Phenylcapric Acid**

$$\text{CH}_3\cdot[\text{CH}_2]_3\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot[\text{CH}_2]_4\cdot\text{COOH}$$

$$\text{C}_{16}\text{H}_{24}\text{O}_2 \quad \text{MW, 248}$$

B.p.  $176-80^\circ/3$  mm.  $D_4^{25}$  0.9817.  $n_D^{20}$  1.5000.

p-Bromophenacyl ester: m.p.  $55.5-56^\circ$ .

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, 54, 2523.

**9-Phenylcapric Acid**

$$\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot[\text{CH}_2]_7\cdot\text{CH}_2\cdot\text{COOH}$$

$$\text{C}_{16}\text{H}_{24}\text{O}_2 \quad \text{MW, 248}$$

Waxy solid. M.p.  $41^\circ$ . B.p.  $228-30^\circ/18$  mm. Very sol. usual org. solvents.

Et ester:  $\text{C}_{18}\text{H}_{28}\text{O}_2$ . MW, 276. B.p.  $220-4^\circ/20$  mm.

Borsche, *Ber.*, 1919, 52, 2085.

**1-Phenylcaproic Acid**

$$\text{CH}_3\cdot[\text{CH}_2]_3\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{COOH}$$

$$\text{C}_{12}\text{H}_{16}\text{O}_2 \quad \text{MW, 192}$$

B.p.  $182-3^\circ/20$  mm.  $D_4^{19}$  1.0225.  $n_D^{19}$  1.5071.

Amide:  $\text{C}_{12}\text{H}_{17}\text{ON}$ . MW, 191. M.p.  $96^\circ$ .

Nitrile:  $\text{C}_{12}\text{H}_{15}\text{N}$ . MW, 173. B.p.  $151.1-152.5^\circ/20$  mm.

Lévy, Jullien, *Bull. soc. chim.*, 1929, 45, 941.

Dolique, *Ann. chim.*, 1931, 15, 468.

**2-Phenylcaproic Acid**

$$\text{CH}_3\cdot[\text{CH}_2]_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}_2\cdot\text{COOH}$$

$$\text{C}_{12}\text{H}_{16}\text{O}_2 \quad \text{MW, 192}$$

d-.

B.p.  $152^\circ/4$  mm.  $D_4^{25}$  1.025.  $n_D$  1.5078.  $[\alpha]_D^{25} + 5.93^\circ$ .

Et ester:  $\text{C}_{14}\text{H}_{20}\text{O}_2$ . MW, 220. B.p.  $123^\circ/2$  mm.  $D_4^{25}$  0.969.  $n_D^{25}$  1.4870.  $[\alpha]_D^{25} + 3.45^\circ$ .

Levene, Marker, *J. Biol. Chem.*, 1931, 93, 765.

**3-Phenylcaproic Acid**

$$\text{CH}_3\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{COOH}$$

$$\text{C}_{13}\text{H}_{18}\text{O}_2 \quad \text{MW, 192}$$

dl-.

Cryst. M.p.  $104.5-105.5^\circ$ . B.p.  $185^\circ/22$  mm.

Chloride:  $\text{C}_{12}\text{H}_{15}\text{OCl}$ . MW, 210.5. B.p.  $138^\circ/18$  mm.

Nitrile:  $\text{C}_{12}\text{H}_{15}\text{N}$ . MW, 173. B.p.  $148-50^\circ/19$  mm.

l-.

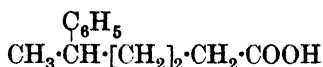
B.p.  $156^\circ/4$  mm.  $[\alpha]_D^{25} - 0.61^\circ$ .

Lévy, *Compt. rend.*, 1933, 197, 772.

Levene, Marker, *J. Biol. Chem.*, 1935, 110, 329.



## 4-Phenylcaproic Acid



$\text{C}_{12}\text{H}_{16}\text{O}_2$  MW, 192

*d.*

B.p. 156°/1 mm.  $D_4^{25}$  1.022.  $[\alpha]_D^{25} + 2.01^\circ$ .

*Et ester*:  $\text{C}_{14}\text{H}_{20}\text{O}_2$ . MW, 220. B.p. 145°/1 mm.  $D_4^{25}$  0.974.  $[\alpha]_D^{25} + 1.40^\circ$ .

*dl.*

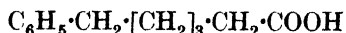
B.p. 173–5°/11 mm.

*Chloride*:  $\text{C}_{12}\text{H}_{15}\text{OCl}$ . MW, 210.5. B.p. 134–6°/13 mm.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 771.

Nenitzescu, Gavăt, *Ann.*, 1935, **519**, 271.

## 5-Phenylcaproic Acid



$\text{C}_{12}\text{H}_{16}\text{O}_2$  MW, 192

Cryst. M.p. 22–4° (11°). B.p. 206–8°/30 mm., 180–90°/17 mm. (186–8°/11 mm.).

*Et ester*:  $\text{C}_{14}\text{H}_{20}\text{O}_2$ . MW, 220. B.p. 162–4°/12 mm.

*Chloride*:  $\text{C}_{12}\text{H}_{15}\text{OCl}$ . MW, 210.5. B.p. 151–2°/11 mm.

*Nitrile*:  $\text{C}_{12}\text{H}_{15}\text{N}$ . MW, 173. B.p. 161–3°/13 mm.

*Anilide*: m.p. 80°.

*p-Toluidide*: m.p. 78°.

*Piperidide*: b.p. 177°/0.03 mm.

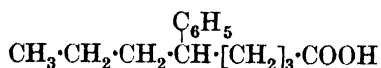
v. Braun, *Ber.*, 1911, **44**, 2873.

Borsche, *Ber.*, 1919, **52**, 2084.

Staudinger, Müller, *Ber.*, 1923, **56**, 714.

Grateau, *Compt. rend.*, 1930, **191**, 947.

## 4-Phenylcaprylic Acid



$\text{C}_{14}\text{H}_{20}\text{O}_2$  MW, 220

*l.*

B.p. 170°/1 mm.  $D_4^{25}$  0.999.  $[\alpha]_D^{25} - 0.82^\circ$ .

*Et ester*:  $\text{C}_{16}\text{H}_{24}\text{O}_2$ . MW, 248. B.p. 152°/1 mm.  $D_4^{25}$  0.958.  $[\alpha]_D^{25} - 0.49^\circ$ .

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 770.

## 7-Phenylcaprylic Acid



$\text{C}_{16}\text{H}_{20}\text{O}_2$  MW, 220

Leaflets. M.p. about 30°. B.p. 209–10°/14 mm.

Borsche, *Ber.*, 1919, **52**, 2084.

Phenylcarbamic Acid (*Carbanilic acid*)

$\text{C}_7\text{H}_7\text{O}_2\text{N}$  MW, 137

Not known in free state.

*Me ester*: phenylurethylan.  $\text{C}_8\text{H}_9\text{O}_2\text{N}$ . MW, 151. Leaflets from EtOH. M.p. 47°.

*Et ester*: see Phenylurethane.

*Propyl ester*:  $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$ . MW, 179. Needles. M.p. 57–9°.

*Isopropyl ester*: needles from EtOH. M.p. 75–6° (90°).

*Butyl ester*:  $\text{C}_{11}\text{H}_{15}\text{O}_2\text{N}$ . MW, 193. Leaflets or prisms from pet. ether. M.p. 65.5° (57°).

*Isobutyl ester*: needles. M.p. 86°.

*tert.-Butyl ester*: needles from pet. ether. M.p. 136°.

*dl-Amly ester*:  $\text{C}_{12}\text{H}_{17}\text{O}_2\text{N}$ . MW, 207. M.p. 31°.

*d-Amly ester*: cryst. from ligroin. M.p. 30°.

*Isoamyl ester*: cryst. from pet. ether. M.p. 55°.

*tert.-Amly ester*: m.p. 42°.

*Allyl ester*: cryst. M.p. 70°.

*l-Menthyl ester*: needles from EtOH. M.p. 111–12°.  $[\alpha]_D^{20} - 77.21^\circ$  in  $\text{CHCl}_3$ .

*dl-Menthyl ester*: needles from MeOH.Aq. M.p. 104°.

*Tropine ester*: see Uretropine.

*Amide*: see Phenylurea.

*Anilide*: see Carbanilide.

*Nitrile*: see Phenylcyanamide.

Jeffreys, *Am. Chem. J.*, 1901, **22**, 18.

Roemer, *Ber.*, 1873, **6**, 1103.

Lambling, *Bull. soc. chim.*, 1898, **19**, 777.

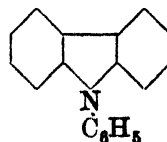
Piccard, Littlebury, *J. Chem. Soc.*, 1912, **101**, 116.

Weizmann, Garrard, *J. Chem. Soc.*, 1920, **117**, 328.

## Phenylcarbazine Acid.

See Phenylhydrazine-β-carboxylic Acid.

## N-Phenylcarbazole



$\text{C}_{16}\text{H}_{18}\text{N}$  MW, 243

Needles or plates from EtOH. M.p. 94–5° (82–4°).

**Picrate:** red needles from ligroin. M.p. 126-9°.

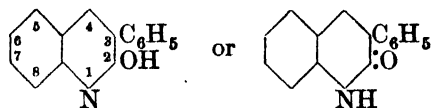
Cassella, D.R.P., 224,951, (*Chem. Zentr.*, 1910, II, 699).

Eckert, Seidel, Endler, *J. prakt. Chem.*, 1922, 104, 88.

Montmollin, Montmollin, *Helv. Chim. Acta*, 1923, 6, 98.

Hager, *Organic Syntheses*, Collective Vol. I, 532 (Note 13).

### 3-Phenylcarbostyryl (2-Hydroxy-3-phenylquinoline)



$C_{15}H_{11}ON$

MW, 221

Needles from EtOH. M.p. 234-5°. Sol. hot EtOH,  $C_6H_6$ . Insol.  $Et_2O$ .

Bischler, Lang, *Ber.*, 1895, 28, 292.

Stoermer, Prigge, *Ann.*, 1915, 409, 27.

Wislicenus, Erbe, *Ann.*, 1920, 421, 146.

### 4-Phenylcarbostyryl (2-Hydroxy-4-phenylquinoline)

Needles. M.p. 259°. Sol. AcOH, hot  $C_6H_6$ . Spar. sol. hot  $H_2O$ , EtOH,  $CHCl_3$ ,  $CS_2$ , ligroin.  $Zn + H \rightarrow$  4-phenylquinoline.

Camps, *Arch. Pharm.*, 1899, 237, 683.

#### Phenyl $\alpha$ -carboxybenzyl Ketone.

See *o*-Phenacylbenzoic Acid.

#### Phenyl-carboxymethyl-glycine.

See 1-Phenyliminodiacetic Acid.

#### Phenyl- $\omega$ -carboxysarcosine.

See 1-Phenyliminodiacetic Acid.

#### Phenylcarbylamine.

See Phenyl isocyanide.

#### Phenylcarbylamine chloride.

See Phenyliminophosgene.

### Phenylchloroacetic Acid (1-Chloro-1-phenylacetic acid)



$C_8H_7O_2Cl$

MW, 170.5

*d*-.  
Cryst. from pet. ether. M.p. 60-1°.  $[\alpha]_D^{20}$  + 191.9° in  $C_6H_6$ .

**Me ester:**  $C_9H_9O_2Cl$ . MW, 184.5. B.p. 178°/40 mm., 124°/8 mm.  $D_4^{20}$  1.1882.  $[\alpha]_D^{18}$  + 25.67°.

**Et ester:**  $C_{10}H_{11}O_2Cl$ . MW, 198.5. B.p. 162°/45 mm., 138°/19 mm., 133°/12 mm.  $D_4^{20}$  1.1594.  $n_D^{20}$  1.5152.  $[\alpha]_D^{20}$  + 121.0° in EtOH.

**Propyl ester:**  $C_{11}H_{13}O_2Cl$ . MW, 212.5.

B.p. 180°/60 mm., 140°/19 mm.  $D_4^{20}$  1.1278.  $n_D^{20}$  1.5095.  $[\alpha]_D^{18}$  + 23.94°.

**Butyl ester:**  $C_{12}H_{15}O_2Cl$ . MW, 226.5. B.p. 163-4°/20 mm.  $D^{20}$  1.1040.

**l-Menthyl ester:** needles from EtOH.Aq. M.p. 56-7°.  $[\alpha]_D^{15.5}$  + 5.7° in EtOH.

**Chloride:**  $C_8H_8OCl_2$ . MW, 189. B.p. 120°/23 mm.  $[\alpha]_D^{18}$  + 158.33° in  $CS_2$ .

*l*-.  
Needles from pet. ether. M.p. 61°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. cold  $H_2O$ .  $[\alpha]_D^{15}$  - 163° in EtOH, - 191.3° in  $C_6H_6$ .

**Me ester:** b.p. 123-6°/11 mm.  $D_4^{18}$  1.213.  $[\alpha]_D^{15}$  - 86.7°.

**Et ester:** b.p. 132-3°/15 mm.  $D_4^{16.4}$  1.162.  $[\alpha]_D^{20}$  - 108° in EtOH.

**l-Menthyl ester:** needles from EtOH.Aq. M.p. 44.5-45.5°.  $[\alpha]_D^{15}$  - 150.1°.

**Chloride:** b.p. 103°/11 mm.  $[\alpha]_{D_{78}}^{20}$  - 250°.

**Dimethylamide:** m.p. 47°.  $[\alpha]_{578} - 110°$  in EtOH.

*dl*-.  
Leaflets. M.p. 78°. Very sol. EtOH,  $Et_2O$ . Mod. sol. hot ligroin. Spar. sol.  $H_2O$ .

**Me ester:** b.p. 248° decomp.

**Et ester:** b.p. 142°/19 mm.

**l-Menthyl ester:** cryst. from  $Et_2O$ . M.p. 28-9°. B.p. 200-1°/12 mm.  $D_4^{20}$  1.064.  $[\alpha]_D^{20}$  - 67.2°.

**Chloride:** b.p. 120°/15 mm., 110°/4 mm.

**Amide:**  $C_8H_8ONCl$ . MW, 169.5. Needles from  $C_6H_6$ . M.p. 116°. Sol. EtOH,  $Et_2O$ .

**Nitrile:**  $C_8H_8NCl$ . MW, 151.5. B.p. 131.5°/13 mm., 108-10°/5 mm.

McKenzie, Clough, *J. Chem. Soc.*, 1909, 95, 784.

Walden, *Ber.*, 1895, 28, 1295.

Bischoff, Walden, *Ann.*, 1894, 279, 122.

Meyer, Boner, *Ann.*, 1883, 220, 44.

Michaël, Jeanprêtre, *Ber.*, 1892, 25, 1680.

Darapsky, *J. prakt. Chem.*, 1919, 99, 188.

McKenzie, Barrow, *J. Chem. Soc.*, 1911, 99, 1916.

McKenzie, Smith, *J. Chem. Soc.*, 1923, 123, 1962.

Freudenberg, Todd, Seidler, *Ann.*, 1933, 501, 217.

Barrow, Thorneycroft, *J. Chem. Soc.*, 1934, 724.

Hignett, Kay, *J. Soc. Chem. Ind.*, 1935, 54, 98t.

#### Phenyl $\omega$ -chlorobenzyl Ketone.

See Desyl chloride.

#### Phenylchloroform.

See Benzotrichloride.

**Phenyl-2-chlorolactic Acid** (2-Chloro-1-hydroxy-2-phenylpropionic acid, cinnamic acid chlorohydrin)



$\text{C}_9\text{H}_9\text{O}_3\text{Cl}$

MW, 200.5

*l.*

Cryst. M.p. 144°.  $[\alpha]_D - 71.7^\circ$ .

*dl.*

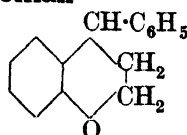
Needles from  $\text{CHCl}_3$ . M.p. 141–2°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin. Hot  $\text{H}_2\text{O} \rightarrow \text{CO}_2 + \text{HCl} + \text{phenylacetaldehyde}$ .  $\text{NaOH} \rightarrow \text{phenylglycidic acid}$ .

Erlenmeyer, *Ann.*, 1892, 271, 150; *Ber.*, 1906, 39, 789.

**2-Phenylchroman.**

See Flavan.

**4-Phenylchroman**



$\text{C}_{15}\text{H}_{14}\text{O}$

MW, 210

Needles from pet. ether. M.p. 38.5°. Addn. of  $\text{FeCl}_3$  to suspension in  $\text{H}_2\text{SO}_4 \rightarrow$  reddish-violet col.

Greenwood, Nierenstein, *J. Chem. Soc.*, 1920, 117, 1597.

**2-Phenylchromanone.**

See Flavanone.

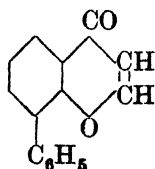
**2-Phenylchromone.**

See Flavone.

**3-Phenylchromone.**

See Isoflavone.

**8-Phenylchromone**



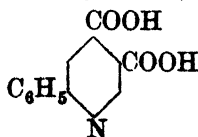
$\text{C}_{15}\text{H}_{10}\text{O}_2$

MW, 222

Needles from EtOH.Aq. M.p. 112°.

Watson, *J. Chem. Soc.*, 1916, 109, 305.

**6-Phenylcinchomeronic Acid** (6-Phenylpyridine-3 : 4-dicarboxylic acid)



$\text{C}_{13}\text{H}_9\text{O}_4\text{N}$

MW, 243

Yellow needles and prisms from  $\text{H}_2\text{O}$ . M.p. 248–50°. Spar. sol.  $\text{H}_2\text{O}$ , Et<sub>2</sub>O. Mod. sol. EtOH.

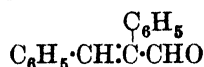
*Di-Me ester*:  $\text{C}_{16}\text{H}_{13}\text{O}_4\text{N}$ . MW, 271. Plates from  $\text{CHCl}_3$ . M.p. 74°.

Boehm, Bournot, *Ber.*, 1915, 48, 1573.

**Phenylcinchoninic Acid.**

See Phenylquinoline-4-carboxylic Acid.

**$\alpha$ -Phenylcinnamaldehyde** (1 : 2-Diphenylacrolein,  $\alpha$ -formylstilbene)



$\text{C}_{15}\text{H}_{12}\text{O}$

MW, 208

Cryst. from EtOH. M.p. 94°. B.p. 195–200°/17 mm.  $\text{Ag}_2\text{O}$  in  $\text{C}_6\text{H}_6 \rightarrow$  1-phenylcinnamic acid.

*Oxime*: leaflets from EtOH. M.p. 165–6°.

*Semicarbazone*: cryst. from EtOH. M.p. 188.5–189.5°.

*Phenylhydrazone*: yellow needles from AcOH. M.p. 125–6°.

Meerwein, *J. prakt. Chem.*, 1918, 97, 281.

Shorugan, Isagulyantz, Machinskaya, *Ber.*, 1933, 66, 389.

**$\beta$ -Phenylcinnamaldehyde** (2 : 2-Diphenylacrolein)



$\text{C}_{15}\text{H}_{12}\text{O}$

MW, 208

Pale yellow prisms from ligroin. M.p. 44°.

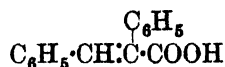
*Semicarbazone*: m.p. 214–15°.

*Phenylhydrazone*: yellow needles. M.p. 173°.

Ziegler, Tiemann, *Ber.*, 1922, 55, 3413.

Kohler, Larsen, *J. Am. Chem. Soc.*, 1935, 57, 1452.

**$\alpha$ -Phenylcinnamic Acid** (Stilbene- $\alpha$ -carboxylic acid, 1 : 2-diphenylacrylic acid)



$\text{C}_{15}\text{H}_{12}\text{O}_2$

MW, 224

*Trans*:

Needles from ligroin or EtOH.Aq. M.p. 172°. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. hot  $\text{H}_2\text{O}$ . Irradiation  $\rightarrow$  *cis*-form.

*Me ester*:  $\text{C}_{16}\text{H}_{14}\text{O}_2$ . MW, 238. Needles from EtOH.Aq. M.p. 77–8°.

*Et ester*:  $\text{C}_{17}\text{H}_{16}\text{O}_2$ . MW, 252. Cryst. M.p. 33–4° (28°). B.p. 214–15°/28 mm., 192°/13 mm.  $D_4^{20} 1.0971$ .  $n_D^{20} 1.5972$ .

1-Menthyl ester: needles or leaflets from EtOH.Aq.  $[\alpha]_D^{20} - 53.44^\circ$  in  $\text{C}_6\text{H}_6$ .

*Phenyl ester*:  $\text{C}_{21}\text{H}_{16}\text{O}_2$ . MW, 300. Needles

from EtOH-CHCl<sub>3</sub>. M.p. 142°. Sol. CHCl<sub>3</sub>. Mod. sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, Et<sub>2</sub>O.

*o*-Tolyl ester: C<sub>22</sub>H<sub>18</sub>O<sub>2</sub>. MW, 314. Needles from EtOH. M.p. 130°.

Amide: C<sub>15</sub>H<sub>13</sub>ON. MW, 223. Needles from Me<sub>2</sub>CO.Aq. M.p. 127°.

Nitrile: C<sub>15</sub>H<sub>11</sub>N. MW, 205. Cryst. from EtOH. M.p. 49-51°. B.p. 213-14°/23 mm.

Anilide: cryst. from EtOH. M.p. 141°.

*Cis*:

Leaflets from EtOH. M.p. 137-8°. More sol. than *trans*-form.

Amide: cryst. from CHCl<sub>3</sub>-ligroin. M.p. 167-8°.

Nitrile: cryst. from EtOH. M.p. 86°. B.p. 228-30°/23 mm.

Anilide: cryst. from CHCl<sub>3</sub>-pet. ether. M.p. 179°.

Stoermer, Voht, *Ann.*, 1915, **409**, 39.

Cabella, *Gazz. chim. ital.*, 1884, **14**, 114.

Rupe, *Ann.*, 1909, **369**, 315.

Walther, *J. prakt. Chem.*, 1896, **53**, 454.

v. Braun, Manz, *Ann.*, 1929, **468**, 258.

Pfeiffer, Engelhardt, Alfuss, *Ann.*, 1928, **467**, 158.

Müller, Gawlich, Krentzmann, *Ann.*, 1935, **515**, 111.

$\beta$ -Phenylcinnamic Acid (2:2-Diphenyl-acrylic acid)



C<sub>15</sub>H<sub>12</sub>O<sub>2</sub> MW, 224

Leaflets from EtOH. M.p. 162°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Spar. sol. H<sub>2</sub>O, ligroin.

Me ester: oil. B.p. 194.6-194.8°/13 mm.

Et ester: b.p. 207°/17 mm.

1-Menthyl ester: needles from EtOH. M.p. 66-7°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> - 37.92° in C<sub>6</sub>H<sub>6</sub>.

Nitrile: pale yellow needles from MeOH. M.p. 49°.

Anilide: m.p. 130-1°.

Anhydride: C<sub>30</sub>H<sub>22</sub>O<sub>3</sub>. MW, 430. Needles from EtOH. M.p. 118-20°. Cold conc. H<sub>2</sub>SO<sub>4</sub> → green col. → red on heating.

de Fazi, *Gazz. chim. ital.*, 1915, **45**, ii, 5.

Rupe, *Ann.*, 1909, **369**, 315; 1913, **395**, 142.

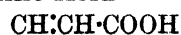
Kohler, Reimer, *Am. Chem. J.*, 1905, **33**, 343.

Rupe, Busolt, *Ber.*, 1907, **40**, 4539.

Posner, *J. prakt. Chem.*, 1910, **82**, 439.

Schlenk, Bergmann, *Ann.*, 1928, **463**, 237.

### *p*-Phenylcinnamic Acid



C<sub>15</sub>H<sub>12</sub>O<sub>2</sub> MW, 224

Needles from 60% AcOH. M.p. 225°.

Et ester: C<sub>17</sub>H<sub>16</sub>O<sub>2</sub>. MW, 252. M.p. 87°.

v. Braun, Nelles, *Ber.*, 1933, **66**, 1465.

Vorländer, *Ber.*, 1935, **68**, 453.

Hey, *J. Chem. Soc.*, 1931, 2478.

### Phenylcitraconic Acid (Benzylmaleic acid)



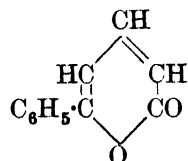
C<sub>11</sub>H<sub>10</sub>O<sub>4</sub> MW, 206

Cryst. from Et<sub>2</sub>O-ligroin. M.p. 105-8°. Very sol. H<sub>2</sub>O, Et<sub>2</sub>O. Mod. sol. CHCl<sub>3</sub>. Insol. CS<sub>2</sub>, ligroin. Hot H<sub>2</sub>O → phenylitaconic acid. 10% NaOH → phenylitaconic + phenyl-atonic acids. Br in sunlight → phenyl-mesaconic acid.

Anhydride: C<sub>11</sub>H<sub>8</sub>O<sub>3</sub>. MW, 188. Prisms from Et<sub>2</sub>O. M.p. 60-1°. Sol. C<sub>6</sub>H<sub>6</sub>, warm Et<sub>2</sub>O, boiling CS<sub>2</sub>. Spar. sol. boiling ligroin. H<sub>2</sub>O → phenylcitraconic acid.

Fittig, Brooke, *Ann.*, 1899, **305**, 21.

### 6-Phenylcoumalin (6-Phenyl- $\alpha$ -pyrone)



C<sub>11</sub>H<sub>8</sub>O<sub>2</sub> MW, 172

Present in Coto bark. Needles from pet. ether or MeOH.Aq. M.p. 68°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH. Mod. sol. warm pet. ether. Picrate: yellow plates from Et<sub>2</sub>O. M.p. 81-2°.

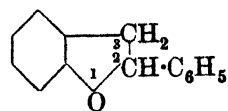
Dimer: cryst. powder. M.p. 219° decomp. Spar. sol. boiling EtOH, Me<sub>2</sub>CO. Insol. Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

Severni, *Gazz. chim. ital.*, 1896, **26**, ii, 338.

Ciamician, Silber, *Ber.*, 1894, **27**, 841.

Kalf, *Rec. trav. chim.*, 1927, **46**, 594.

### 2-Phenylcoumaran



C<sub>14</sub>H<sub>12</sub>O

MW, 196

Cryst. M.p. 32–3°. Volatile in steam.  
Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  pale yellow col.

Stoermer, Reuter, *Ber.*, 1903, **36**, 3983.

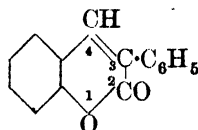
### 3-Phenylcoumaran.

Needles from EtOH. M.p. 38.5°. B.p. 167°/14 mm. Sol. in conc.  $\text{H}_2\text{SO}_4$  turns yellow on standing. Conc.  $\text{H}_2\text{SO}_4 + \text{FeCl}_3 \rightarrow$  brownish-green col.

Stoermer, Reuter, *Ber.*, 1903, **36**, 3984.

Stoermer, Kippe, *Ber.*, 1903, **36**, 4006, 4008.

### 3-Phenylcoumarin



$\text{C}_{15}\text{H}_{10}\text{O}_2$  MW, 222

Needles from AcOH. M.p. 140–1°.

Semicarbazone: pale yellow cryst. Decomp. at 210–13°.

Lovett, Roberts, *J. Chem. Soc.*, 1928, 1977.

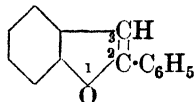
Stoermer, Prigge, *Ann.*, 1915, **409**, 27.

### 4-Phenylcoumarin.

Needles from EtOH or  $\text{H}_2\text{O}$ . M.p. 105°. Sol. most org. solvents. Spar. sol. boiling  $\text{H}_2\text{O}$ . Sol. conc.  $\text{H}_2\text{SO}_4$ .

Stoermer, Friderici, *Ber.*, 1908, **41**, 340.

### 2-Phenylcoumarone



$\text{C}_{14}\text{H}_{10}\text{O}$  MW, 194

Leaflets from EtOH.Aq. M.p. 120–1°. Volatile in steam. Sol. conc.  $\text{H}_2\text{SO}_4$  with yellow col. showing blue fluor. on heating.

Stoermer, Reuter, *Ber.*, 1903, **36**, 3981.

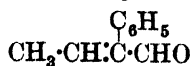
v. Kostanecki, Tambor, *Ber.*, 1909, **42**, 826.

### 3-Phenylcoumarone.

Cryst. M.p. 42°. B.p. 316–17°, 177–8°/15 mm.  $D_{20}^{20}$  1.449. Sol. conc.  $\text{H}_2\text{SO}_4$  with orange-yellow col. showing blue fluor. on heating.

Stoermer, Kippe, *Ber.*, 1903, **36**, 4004.

### 1-Phenylcrotonaldehyde



$\text{C}_{10}\text{H}_{10}\text{O}$

MW, 146

B.p. 117°/15 mm.  $D_{20}^{20}$  1.045.  $n_D^{20}$  1.5605.

Oxime: m.p. 116°.

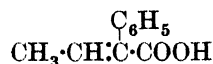
Semicarbazone: m.p. 201°.

Tiffeneau, Weill, *Compt. rend.*, 1935, **200**, 1217.

### 2-Phenylcrotonaldehyde.

$\beta$ -Methylcinnamaldehyde, *q.v.*

1-Phenylcrotonic Acid ( $\alpha$ -Ethylidenepherylacetic acid,  $\beta$ -methylatropic acid)



$\text{C}_{10}\text{H}_{10}\text{O}_2$  MW, 162

Prisms from EtOH, needles or plates from  $\text{H}_2\text{O}$ . M.p. 136°. Spar. sol. cold  $\text{H}_2\text{O}$ .  $\text{KMnO}_4 \rightarrow$  benzoylformic acid + acetaldehyde.

Et ester:  $\text{C}_{12}\text{H}_{14}\text{O}_2$ . MW, 190. B.p. 128–31°/15 mm. Volatile in steam.

1-Menthyl ester: yellow oil. Decomp. on dist.  $[\alpha]_D^{20} - 46.13^\circ$  in  $\text{C}_6\text{H}_6$ .

Amide:  $\text{C}_{10}\text{H}_{11}\text{ON}$ . MW, 161. Plates from  $\text{C}_6\text{H}_6$ . M.p. 98–9°.

Nitrile:  $\text{C}_{10}\text{H}_9\text{N}$ . MW, 143. B.p. 224–6°/751 mm., 125°/13–14 mm.  $D_4^{20}$  1.013.  $n_D^{20}$  1.5555.

Rupe, Busolt, *Ann.*, 1909, **369**, 332.

Dimroth, Feuchter, *Ber.*, 1903, **36**, 2253.

Knowles, Cloke, *J. Am. Chem. Soc.*, 1932, **54**, 2028.

Pfeiffer, Engelhardt, Alfuss, *Ann.*, 1928, **467**, 158.

Ray, *Chem. Abstracts*, 1928, **22**, 3647.

### 2-Phenylcrotonic Acid.

See  $\beta$ -Methylcinnamic Acid.

3-Phenylcrotonic Acid (2-Benzylacrylic acid)



$\text{C}_{10}\text{H}_{10}\text{O}_2$  MW, 162

Two forms. (i) Plates from  $\text{C}_6\text{H}_6$ , pearly leaflets from  $\text{H}_2\text{O}$ . M.p. 65°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ . (ii) Liq. Passes into (i) on heating with dil. HCl.

Bougault, *Compt. rend.*, 1917, **164**, 635.

Fittig, Luib, *Ann.*, 1894, **283**, 55, 302.

Vorländer, Strunck, *Ann.*, 1906, **345**, 244.

### 1-Phenylcrotonyl Alcohol.

See Propenylphenylcarbinol.

Phenylcyanamide (Cyananilide, phenylcarbamic nitrile, N-cyanoaniline)



$\text{C}_7\text{H}_6\text{N}_2$

MW, 118

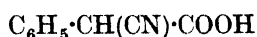
Cryst. from  $\text{H}_2\text{O}$  or  $\text{Et}_2\text{O}$ . M.p.  $47^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ . Sol.  $\text{KOH}$ . Aq.

Berger, *Monatsh.*, 1884, **5**, 219.

### Phenyl cyanide.

See Benzonitrile.

**Phenylcyanoacetic Acid** ( $\alpha$ -Cyanophenylacetic acid, phenylmalonic mononitrile)



$\text{C}_9\text{H}_7\text{O}_2\text{N}$  MW, 161

Cryst. from  $\text{Et}_2\text{O}$ -ligroin. M.p.  $92^\circ$ . Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , hot  $\text{C}_6\text{H}_6$ . Insol. ligroin. At  $150-60^\circ \rightarrow$  benzyl cyanide.

*Et ester*:  $\text{C}_{11}\text{H}_{11}\text{O}_2\text{N}$ . MW, 189. Oil. B.p.  $275^\circ$  slight decomp.,  $165.5^\circ/20$  mm. Misc. with org. solvents. Insol.  $\text{H}_2\text{O}$ .

*Amide*:  $\text{C}_9\text{H}_8\text{ON}_2$ . MW, 160. Cryst. from  $\text{EtOH}$ . M.p.  $147^\circ$ . Sol. hot  $\text{EtOH}$ . Insol.  $\text{H}_2\text{O}$ .

*Methylamide*:  $\text{C}_{10}\text{H}_{10}\text{ON}_2$ . MW, 174. M.p.  $102^\circ$ .

*Anilide*: needles from  $\text{EtOH}$ . M.p.  $136^\circ$ . Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

*o-Toluidide*: m.p.  $139^\circ$ .

*m-Toluidide*: m.p.  $131^\circ$ .

*p-Toluidide*: needles from  $\text{EtOH}$ . M.p.  $139^\circ$ .

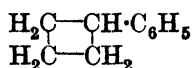
Merck, D.R.P., 606,349, (*Chem. Zentr.*, 1935, I, 2048).

Heseler, *Am. Chem. J.*, 1904, **32**, 122.

### 2-Phenyl-3-cyanocinchoninic Acid.

See under 2-Phenylquinoline-3:4-dicarboxylic Acid.

### Phenylcyclobutane (Cyclobutylbenzene)

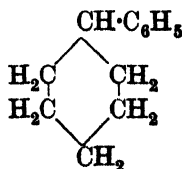


$\text{C}_{10}\text{H}_{12}$  MW, 132

B.p.  $190-1^\circ/755$  mm.,  $101-2^\circ/41$  mm.  $D_4^{20}$  0.9378.  $n_D^{20}$  1.5277.

Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.

**Phenylcyclohexane** (Cyclohexylbenzene, hexahydrodiphenyl)



$\text{C}_{12}\text{H}_{16}$  MW, 160

Plates. M.p.  $7-8^\circ$ . B.p.  $235-6^\circ$ ,  $156^\circ/80$  mm.,  $127-8^\circ/30$  mm.,  $106^\circ/12$  mm.  $D_4^{20}$  0.9502.  $n_D^{20}$  1.5329. Stable to cold  $\text{KMnO}_4$ .

Meerwein, *Ann.*, 1919, **419**, 171.

Bedos, *Compt. rend.*, 1923, **177**, 111.

Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.

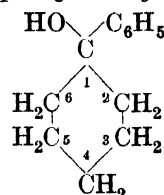
Neunhoffer, *J. prakt. Chem.*, 1932, **133**, 95.

Truffault, *Bull. soc. chim.*, 1934, **1**, 391.

### Phenylcyclohexane-carboxylic Acid.

See Phenylhexahydrobenzoic Acid.

**1-Phenylcyclohexanol** (1-Hydroxyhexahydrodiphenyl, 1-phenylhexahydrophenol)



$\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176

Prisms from ligroin. M.p.  $63-63.5^\circ$ . B.p.  $156.5-158.5^\circ/28$  mm.,  $141-4^\circ/14$  mm. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin. Insol.  $\text{H}_2\text{O}$ .

Auwers, Treppmann, *Ber.*, 1915, **48**, 1216.

Sabatier, Mailhe, *Compt. rend.*, 1904, **138**, 1322.

Kurssanow, *Chem. Zentr.*, 1907, I, 1744.

**2-Phenylcyclohexanol** (2-Hydroxyhexahydrodiphenyl, 2-phenylhexahydrophenol).

Cryst. from pet. ether. M.p.  $56-7^\circ$ . B.p.  $153-4^\circ/16$  mm.

**3:5-Dinitrobenzoyl**: cryst. from  $\text{EtOH}$ . M.p.  $121-121.5^\circ$ .

*Phenylurethane*: m.p.  $137-138.5^\circ$ .

*Phthalate*: m.p.  $185-6^\circ$ .

Bedos, *Bull. soc. chim.*, 1926, **39**, 292.

Cook, Hewett, Lawrence, *J. Chem. Soc.*, 1936, 75.

**3-Phenylcyclohexanol** (3-Hydroxyhexahydrodiphenyl, 3-phenylhexahydrophenol).

Needles from pet. ether. M.p.  $81^\circ$  ( $79.5-80.5^\circ$ ). Sol. most org. solvents. Volatile in steam. Odour resembles geranium.

*Acetyl*: plates from  $\text{EtOH}$ . M.p.  $43-4^\circ$ . B.p.  $300^\circ$ .

*Benzoyl*: prisms from  $\text{EtOH}$ . M.p.  $68^\circ$ .

*o-Nitrobenzoyl*: needles from  $\text{EtOH}$ . M.p.  $70^\circ$ .

Boyd, Clifford, Probert, *J. Chem. Soc.*, 1920, **117**, 1383.

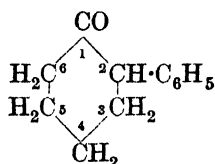
Crossley, Renouf, *J. Chem. Soc.*, 1915, **107**, 608.

**4-Phenylcyclohexanol** (4-Hydroxyhexahydrodiphenyl, 4-phenylhexahydrophenol).

Needles from  $C_6H_6$ . M.p. 132–3°. B.p. 155–8°/13 mm. Very sol.  $Et_2O$ . Mod. sol. ligroin. Spar. sol.  $H_2O$ .

Kurssanow, *Ann.*, 1901, **318**, 325; *Chem. Zentr.*, 1923, III, 1075.

Wuyts, *Chem. Zentr.*, 1912, II, 1006.

**2-Phenylcyclohexanone**

$C_{12}H_{14}O$

MW, 174

Cryst. from pet. ether or  $EtOH$ . M.p. 63° (50–3°). B.p. 180°/16 mm. Spar. sol.  $H_2O$ .

Does not form bisulphite comp.

Oxime: m.p. 169°.

Semicarbazone: m.p. 190°.

Le Brazidec, *Compt. rend.*, 1914, **159**, 775.

v. Braun, Gruber, Kirschbaum, *Ber.*, 1922, **55**, 3670.

Levy, Stiras, *Bull. soc. chim.*, 1931, **49**, 1830.

Cook, Hewett, Lawrence, *J. Chem. Soc.*, 1936, 76.

Sherwood, Short, Woodcock, *ibid.*, 323.

**3-Phenylcyclohexanone.**

Oil. B.p. 287–8°/736 mm., 169–169.5°/18 mm. Alc.  $H_2SO_4 \rightarrow$  reddish-yellow sol. with green fluor.

Oxime: plates from  $EtOH$ . M.p. 128–9°. Sol. most org. solvents. Mod. sol.  $Et_2O$ . Insol. pet. ether.

Semicarbazone: prisms from  $EtOH$ . M.p. 167°. Sol.  $MeOH$ ,  $EtOH$ ,  $Me_2CO$ ,  $CHCl_3$ . Mod. sol.  $Et_2O$ .

Boyd, Clifford, Probert, *J. Chem. Soc.*, 1920, **117**, 1383.

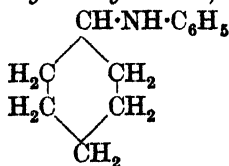
**4-Phenylcyclohexanone.**

Cryst. from pet. ether. M.p. 78°.

Semicarbazone: cryst. from  $EtOH$ . M.p. 229° decomp.

v. Braun, Weissbach, *Ber.*, 1931, **64**, 1788.

**N-Phenylcyclohexylamine** (Hexahydrodiphenylamine, N-cyclohexylaniline)



$C_{12}H_{17}N$

MW, 175

M.p. 14°. B.p. 191–2°/73 mm., 180.5–182.5°/66 mm., 134–5°/6 mm. Sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

$B, HCl$ : needles. M.p. 230.5° (204–5°). Sol.  $EtOH$ . Spar. sol.  $Et_2O$ .

$B_2, H_2SO_4$ : needles. M.p. 226–227.5°.

$B, HBr$ : needles. M.p. 184°.

$B, HI$ : needles. M.p. 176°.

$B, HNO_3$ : m.p. 173°.

Acetate: needles. M.p. 154–155.5°.

N-Acetyl: m.p. 69–70°. Sol.  $EtOH$ . Insol.  $H_2O$ .

N-p-Toluenesulphonyl: m.p. 141–2°.

$B_2, H_2PtCl_6$ : m.p. 177°.

Picrate: yellow prisms. M.p. 164–5°.

Kurssanow, *Chem. Zentr.*, 1907, I, 1744.

Guyot, Fournier, *Bull. soc. chim.*, 1930, **47**, 203.

Skita, Keil, *Ber.*, 1928, **61**, 1682.

Bucherer, Fischbeck, *J. prakt. Chem.*, 1934, **140**, 69.

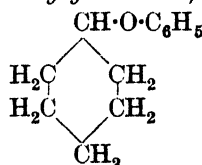
Hickinbottom, *J. Chem. Soc.*, 1932, 2646.

Hiers, Adams, *J. Am. Chem. Soc.*, 1927, **49**, 1102.

I.G., D.R.P., 483,205, (*Chem. Zentr.*, 1929, II, 2938).

Forge, *Ann. chim.*, 1921, **15**, 291.

**Phenyl cyclohexyl Ether** (Hexahydrodiphenyl ether, phenoxycyclohexane)



$C_{12}H_{16}O$

MW, 176

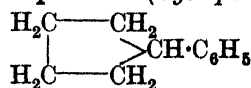
B.p. 260–2°, 140°/21.5 mm.  $D_4^{20}$  1.0241,  $D_4^{20}$  1.0077.

Kurssanow, *J. Russ. Phy.-Chem. Soc.*, 1916, **48**, 1172.

Scrath, Quasebath, *Ber.*, 1924, **57**, 854.

v. Duzee, Adkins, *J. Am. Chem. Soc.*, 1935, **57**, 147.

**Phenylcyclopentane** (Cyclopentylbenzene)



$C_{11}H_{14}$

MW, 146

B.p. 213–15°, 116–17°/37 mm.  $D_4^{20}$  0.9502,  $D_4^{20}$  0.9432.  $n_D^{20}$  1.5309.

Zelinski, *Ber.*, 1925, **58**, 2755.

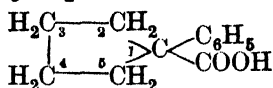
v. Braun, Kühn, *Ber.*, 1927, **60**, 2561.

Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.

Dupont, *Chem. Zentr.*, 1936, II, 613.

**1-Phenylcyclopentane-1-carboxylic Acid**

399

**1-Phenylcyclopentane-1-carboxylic Acid** $C_{12}H_{14}O_2$ 

MW, 190

M.p. 158-9°.

Amide:  $C_{12}H_{15}ON$ . MW, 189. Cryst. from  $C_6H_6$ . M.p. 157-8°.

See third reference above.

**2-Phenylcyclopentane-1-carboxylic Acid.**

Thick oil. B.p. 190-2°/13 mm.

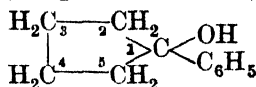
Chloride:  $C_{12}H_{13}OCl$ . MW, 208.5. B.p. 150-3°/12 mm.Anilide: cryst. from  $Et_2O$ . M.p. 93-5°.v. Braun, Kühn, *Ber.*, 1927, **60**, 2560.**3-Phenylcyclopentane-1-carboxylic Acid.**

B.p. 196-8°/5 mm.

Chloride: b.p. 159-62°/15 mm.

Amide:  $C_{12}H_{15}ON$ . MW, 189. M.p. 149°.

Anilide: m.p. 107°.

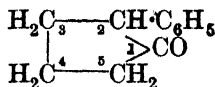
Nenitzescu, Gavât, *Ann.*, 1935, **519**, 268.**1-Phenylcyclopentanol** $C_{11}H_{14}O$ 

MW, 162

B.p. 132-3°/18 mm., 121.0-121.1°/6 mm.  $D_4^{20}$  1.0530.  $n_D^{20}$  1.5479.Zelinski, *Ber.*, 1925, **58**, 2755.Dupont, Chavanne, *Bull. soc. chim. Belg.*, 1933, **42**, 537.**3-Phenylcyclopentanol.**

B.p. 155-6°/10 mm.

Acetyl: thick oil. B.p. 154°/12 mm.

Borsche, Menz, *Ber.*, 1908, **41**, 203.**2-Phenylcyclopentanone** $C_{11}H_{12}O$ 

MW, 160

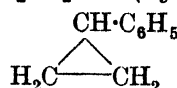
M.p. 126-7°.

Oxime: m.p. 146° decomp.

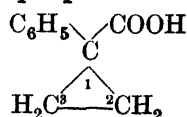
Semicarbazone: m.p. 228° decomp.

Mitchovitch, *Compt. rend.*, 1935, **200**, 1601.**3-Phenylcyclopentanone.**

B.p. 154-5°/10 mm.

Semicarbazone: leaflets from  $EtOH$ . M.p. 181° decomp.Borsche, Menz, *Ber.*, 1908, **41**, 204.**3-Phenylcyclopropane-1 : 2-dicarboxylic Acid****Phenylcyclopropane (Cyclopropylbenzene)** $C_9H_{10}$ 

MW, 118

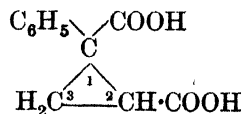
B.p. 173.6°/758 mm., 79-80°/37 mm.  $D_4^{20}$  0.9317.  $n_D^{20}$  1.5285. 60%  $H_2SO_4 \rightarrow$  dimer.Dimer: thick oil. B.p. 330-2°/750 mm.  $D_0^{17}$  1.002.  $n_D^{17}$  1.5710.Kishner, *Chem. Zentr.*, 1913, II, 2129.Lespieau, *Compt. rend.*, 1930, **190**, 1129.Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.**1-Phenylcyclopropane-1-carboxylic Acid** $C_{10}H_{10}O_2$ 

MW, 162

M.p. 86-7°.

Amide:  $C_{10}H_{11}ON$ . MW, 161. M.p. 100-1°.Nitrile:  $C_{10}H_9N$ . MW, 143. B.p. 250-3°/751 mm., 98-100°/1 mm.  $D_4^{20}$  1.0156.  $n_D^{20}$  1.3676.Knowles, Cloke, *J. Am. Chem. Soc.*, 1932, **54**, 2028; *Chem. Abstracts*, 1935, **29**, 2156.

See also last reference above.

**2 - Phenylcyclopropane - 1 - carboxylic Acid.**Needles from  $H_2O$ . M.p. 105°.Et ester:  $C_{12}H_{14}O_2$ . MW, 190. Needles from ligroin. M.p. 39°. B.p. 144-8°/15 mm.Amide: leaflets from hot  $H_2O$ . M.p. 187-8°.Buchner, Geronimus, *Ber.*, 1903, **36**, 3783.**1-Phenylcyclopropane-1 : 2-dicarboxylic Acid** $C_{11}H_{10}O_4$ 

MW, 206

Free acid unstable.

Anhydride:  $C_{22}H_{18}O_7$ . MW, 394. Needles from  $EtOH$ . Aq. M.p. 99°. Sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ . Sol. hot  $H_2O$  with acid reaction.Ruhemann, *J. Chem. Soc.*, 1902, **81**, 1215.**3-Phenylcyclopropane-1 : 2-dicarboxylic Acid.**

Cis :

Prisms from  $EtOH$ . M.p. 175°. Sol.  $EtOH$ ,



Et<sub>2</sub>O. Less sol. H<sub>2</sub>O. Heat in vacuo → anhydride.

*Di-Me ester* : C<sub>13</sub>H<sub>14</sub>O<sub>4</sub>. MW, 234. Needles from ligroin. M.p. 63°. B.p. 200–14°/20 mm.

*Di-Et ester* : C<sub>15</sub>H<sub>18</sub>O<sub>4</sub>. MW, 262. Viscous oil. B.p. 256–7°/120 mm.

*Anhydride* : cryst. from boiling Et<sub>2</sub>O. M.p. 134°. B.p. 282°/190 mm.

*Trans* :

Needles from H<sub>2</sub>O. M.p. 121°.

Buchner, Dessauer, *Ber.*, 1892, 25, 1147.

Buchner, Perkel, *Ber.*, 1903, 36, 3777.

Buchner, *Ber.*, 1888, 21, 2645.

Haerdi, Thorpe, *J. Chem. Soc.*, 1925, 1243.

### N-Phenyldiacetamide.

See Diacetanilide.

### Phenyl-diaminoditolyl-methane.

See Diaminodimethyltriphenylmethane.

### N-Phenyldibenzamide.

See Dibenzanilide.

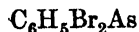
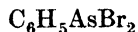
### 2-Phenyl-1 : 3-dibenzoylpropane.

See Benzylidene-diacetophenone.

### Phenyldibenzylamine.

See Dibenzylaniline.

### Phenyldibromoarsine (Dibromo-phenyl-arsine, phenylarsen-dibromide)



MW, 312

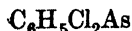
B.p. 285° decomp. D<sup>15</sup> 2-0983.

La Coste, Michaelis, *Ann.*, 1880, 201, 203.

Winmill, *J. Chem. Soc.*, 1912, 101, 723.

Roeder, Blasi, *Ber.*, 1914, 47, 2752.

### Phenyldichloroarsine (Dichloro-phenyl-arsine, phenylarsen-dichloride)



MW, 223

B.p. 252–5°.

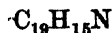
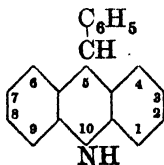
La Coste, Michaelis, *Ann.*, 1880, 201, 193.

Winmill, *J. Chem. Soc.*, 1912, 101, 720.

### Phenyl ω-dichlorobenzyl Ketone.

See α-Dichlorodeoxybenzoin.

### 5-Phenyl-5 : 10-dihydroacridine



MW, 257

Needles from propyl alcohol or C<sub>6</sub>H<sub>6</sub>. M.p. 170° (163°). Mod. sol. Et<sub>2</sub>O, hot EtOH.

*N-Me* : C<sub>20</sub>H<sub>17</sub>N. MW, 271. Needles. M.p. 104°.

*N-Acetyl* : cryst. from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 128°.

Bernthsen, *Ann.*, 1884, 224, 25.

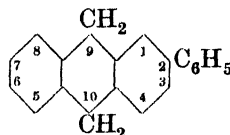
Bergmann, Blum-Bergmann, v. Christiani, *Ann.*, 1930, 483, 85.

### 10-Phenyl-5 : 10-dihydroacridine (N-Phenyldihydroacridine).

Needles or prisms from AcOH. M.p. 119°. Very sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Sol. EtOH. Insol. H<sub>2</sub>O. Alc. sol. shows blue fluor.

Ullmann, Maag, *Ber.*, 1907, 40, 2518.

### 2-Phenyl-9 : 10-dihydroanthracene



MW, 256

Yellowish cryst. from AcOH. M.p. 93–6°. Sol. CHCl<sub>3</sub>, Me<sub>2</sub>CO, CCl<sub>4</sub>, CS<sub>2</sub>. Mod. sol. Et<sub>2</sub>O, AcOH. CrO<sub>3</sub> in boiling AcOH → 2-phenyl-anthraquinone.

Scholl, Neovius, *Ber.*, 1911, 44, 1081.

### 9-Phenyl-9 : 10-dihydroanthracene.

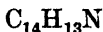
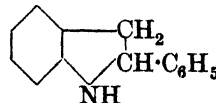
Needles from MeOH. M.p. 91–91.5°.

Haack, *Ber.*, 1929, 62, 1783.

### 2-Phenyl-2 : 3-dihydrobenz-γ-pyrone.

See Flavanone.

### 2-Phenyl-2 : 3-dihydroindole (2-Phenyl-indoline)



MW, 195

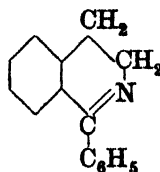
Cryst. from ligroin. M.p. 46°. Sol. dil. min. acids. Volatile in steam.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub> : yellowish-red needles. M.p. 191° decomp. Insol. EtOH.

Pictet, *Ber.*, 1886, 19, 1065.

Stoermer, *Ber.*, 1898, 31, 2540.

### 1-Phenyl-3 : 4-dihydroisoquinoline



MW, 207

Prisms from pet. ether. M.p. 73–4°. B.p. 320°/718 mm., 194–6°/23 mm. Spar. sol. usual org. solvents. Insol. H<sub>2</sub>O.

*B, HCl*: needles. M.p. 225°. Sol. H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O, pet. ether.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: orange prisms from H<sub>2</sub>O. M.p. 230–3°.

*Picrate*: yellow leaflets from CHCl<sub>3</sub>-Et<sub>2</sub>O. M.p. 173–5° (163°). Mod. sol. hot H<sub>2</sub>O.

Pictet, Kay, *Ber.*, 1909, **42**, 1975.

Späth, Berger, Kuntara, *Ber.*, 1930, **63**, 139.

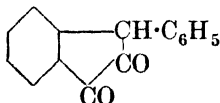
### 3-Phenyl-3 : 4-dihydroquinazoline.

See Orexine.

### Phenyl 2 : 4-dihydroxyphenacyl Ether.

See under Fisetol.

### 3-Phenyl-1 : 2-diketohydrindene (3-Phenyl-indandione-1 : 2)



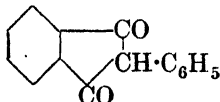
C<sub>15</sub>H<sub>10</sub>O<sub>2</sub> MW, 222

Needles from ligroin. M.p. 137–8°.

*Disemicarbazone*: yellow needles from EtOH. M.p. 252° decomp.

Pfeiffer, Waal, *Ann.*, 1935, **520**, 192.

### 2-Phenyl-1 : 3-diketohydrindene (2-Phenyl-indandione-1 : 3)



C<sub>15</sub>H<sub>10</sub>O<sub>2</sub> MW, 222

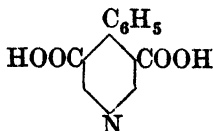
Leaflets from EtOH. M.p. 147–8° (145°). Sol. usual org. solvents. Insol. H<sub>2</sub>O. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with blue col. Sol. alkalis with deep red col.

*Dioxime*: needles from EtOH. M.p. 193–6°.

Nathanson, *Ber.*, 1893, **28**, 2576.

Hantzsch, Gajewski, *Ann.*, 1912, **392**, 303.

### 4-Phenyldinicotinic Acid (4-Phenylpyridine-3 : 5-dicarboxylic acid)



C<sub>13</sub>H<sub>9</sub>O<sub>4</sub>N MW, 243

Greenish-yellow plates + ½ H<sub>2</sub>O. M.p. 229–30°, anhyd. 245–6° decomp.

Weber, *Ann.*, 1887, **241**, 13.

Dict. of Org. Comp.—III.

### Phenyldiphenacylmethane.

See Benzylidene-diacetophenone.

### Phenyl-diphenyl.

See Diphenylbenzene.

### Phenyldiphenylenemethane.

See 9-Phenylfluorene.

### Phenyldiphenylmethane.

See *p*-Benzylidiphenyl.

### Phenyldithiocarbamic Acid (Dithiocarbamic acid)



C<sub>7</sub>H<sub>7</sub>NS<sub>2</sub> MW, 169

*NH<sub>4</sub> salt*: yellow prisms. M.p. 108° decomp. Sol. 4 parts H<sub>2</sub>O at 35°. Spar. sol. EtOH.

*Me ester*: phenyldithiourethylan. C<sub>8</sub>H<sub>9</sub>NS<sub>2</sub>. MW, 183. Leaflets from EtOH. Aq. M.p. 95–6° (87–8°).

*Et ester*: see Phenyldithiourethane.

*Propyl ester*: C<sub>10</sub>H<sub>13</sub>NS<sub>2</sub>. MW, 211. Cryst. from pet. ether. M.p. 66–7°.

*Allyl ester*: C<sub>10</sub>H<sub>11</sub>NS<sub>2</sub>. MW, 209. M.p. 42°.

*Isoamyl ester*: C<sub>12</sub>H<sub>17</sub>NS<sub>2</sub>. MW, 239. Plates. M.p. 71°.

*Phenyl ester*: C<sub>13</sub>H<sub>11</sub>NS<sub>2</sub>. MW, 245. Cryst. from EtOH. M.p. 104–6° decomp.

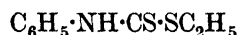
*Benzyl ester*: C<sub>14</sub>H<sub>13</sub>NS<sub>2</sub>. MW, 259. Cryst. from EtOH. M.p. 84–5°.

Freund, Bachrach, *Ann.*, 1895, **285**, 199.

v. Braun, *Ber.*, 1902, **35**, 3384.

Roshdestwenski, *Chem. Zentr.*, 1910, **I**, 910.

### Phenyldithiourethane (Dithiocarbamic acid ethyl ester)



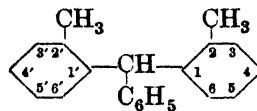
C<sub>9</sub>H<sub>11</sub>NS<sub>2</sub> MW, 197

Plates from EtOH. M.p. 60–1°. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

Will, *Ber.*, 1882, **15**, 1305.

Losanitsch, *Ber.*, 1891, **24**, 3025.

### Phenyldi-*o*-tolylmethane (2 : 2'-Dimethyltriphenylmethane)



C<sub>21</sub>H<sub>20</sub> MW, 272

Cryst. from AcOH. M.p. 104°. B.p. 180–5°/12 mm.

Weiss, Reichel, *Monatsh.*, 1929, **53**, 197.

### Phenyldi-*p*-tolylmethane (4 : 4'-Dimethyltriphenylmethane).

Needles from MeOH. M.p. 55–6° (52°). Very

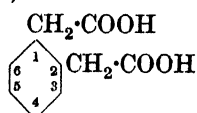
sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Less sol. EtOH, AcOH, ligroin.

Kliegl, *Ber.*, 1905, **38**, 85.

Guyot, Kovache, *Compt. rend.*, 1912, **154**, 122.

Vorländer, *Ber.*, 1911, **44**, 2470.

**o-Phenylenediacetic Acid** (o-Xylene- $\omega$  :  $\omega'$ -dicarboxylic acid)



C<sub>10</sub>H<sub>10</sub>O<sub>4</sub> MW, 194

Needles from H<sub>2</sub>O or Et<sub>2</sub>O. M.p. 150° (148-5-149°). Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O.  $k = 1.111 \times 10^{-4}$  at 25°.

*Di-Ester*: C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>. MW, 250. Oil. B.p. 185°/15 mm.

*Diamide*: C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 192. Needles from H<sub>2</sub>O. M.p. 198°. Spar. sol. EtOH.

*Dinitrile*: o-xylylene dicyanide,  $\omega$  :  $\omega'$ -dicyano-o-xylene. C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>. MW, 156. Exists in two forms. (i) Labile. Prisms or needles from MeOH. M.p. 18°. (ii) Stable. Prisms from EtOH. M.p. 60°. Sol. EtOH, Et<sub>2</sub>O.

Moore, Thorpe, *J. Chem. Soc.*, 1908, **93**, 175.

Baeyer, Pape, *Ber.*, 1884, **17**, 447.

v. Braun, Kruber, Danziger, *Ber.*, 1916, **49**, 2648.

**m-Phenylenediacetic Acid** (m-Xylene- $\omega$  :  $\omega'$ -dicarboxylic acid).

Needles from H<sub>2</sub>O. M.p. 170°.

*Di-Me ester*: C<sub>12</sub>H<sub>14</sub>O<sub>4</sub>. MW, 222. B.p. 298-300°, 185-7°/15 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>.

*Di-Et ester*: b.p. 188-9°/12 mm.

*Dinitrile*: m-xylylene dicyanide,  $\omega$  :  $\omega'$ -dicyano-m-xylene. Cryst. M.p. 28-9°. B.p. 305-10°/300 mm. part. decomp., 170°/20-30 mm. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, ligroin.

Kipping, Oddo, *J. Chem. Soc.*, 1888, **53**, 41.

Oddo, Gazz. chim. ital., 1893, **23**, ii, 337. Titley, *J. Chem. Soc.*, 1928, 2579.

**p-Phenylenediacetic Acid** (p-Xylene- $\omega$  :  $\omega'$ -dicarboxylic acid).

Needles from H<sub>2</sub>O. M.p. 244° (236°). Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>, CS<sub>2</sub>, pet. ether.

*Di-Me ester*: leaflets. M.p. 56.5-57°. B.p. 189-90°/15 mm.

*Di-Et ester*: m.p. 59°.

*Diamide*: leaflets or needles from H<sub>2</sub>O. Does not melt below 290°. Spar. sol. usual solvents.

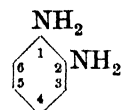
*Dinitrile*: p-xylylene dicyanide,  $\omega$  :  $\omega'$ -dicyano-p-xylene. Needles from H<sub>2</sub>O, prisms from Et<sub>2</sub>O. M.p. 98°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Mod. sol. hot H<sub>2</sub>O.

See last reference above and also

Zincke, Klippert, *Ber.*, 1876, **9**, 1767.

Kipping, *J. Chem. Soc.*, 1888, **53**, 44.

**o-Phenylenediamine** (1 : 2-Diaminobenzene)



C<sub>6</sub>H<sub>8</sub>N<sub>2</sub> MW, 108

Leaflets from H<sub>2</sub>O, plates from CHCl<sub>3</sub>. M.p. 102-3°. B.p. 256-8°/760 mm. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Sol. hot H<sub>2</sub>O.  $k = 3.3 \times 10^{-10}$  at 25°.

N-Me : see N-Methyl-o-phenylenediamine.

N-Di-Me : see o-Aminodimethylaniline.

N : N'-Di-Me : see sym.-Dimethyl-o-phenylenediamine.

N-Et : see N-Ethyl-o-phenylenediamine.

N-Di-Et : see o-Aminodiethylaniline.

N-Phenyl : see o-Aminodiphenylamine.

N-Diphenyl : see o-Aminotriphenylamine.

N : N'-Diphenyl : see sym.-Diphenyl-o-phenylenediamine.

N : N'-Tetra-Me : C<sub>10</sub>H<sub>16</sub>N<sub>2</sub>. MW, 164. Oil. B.p. 215-18°/735 mm. Hot FeCl<sub>3</sub>  $\rightarrow$  red col. B<sub>2</sub>HCl : prisms. M.p. 180°.

N-p-Tolyl : see 2'-Amino-4-methyldiphenylamine.

N-Dibenzyl : C<sub>20</sub>H<sub>20</sub>N<sub>2</sub>. MW, 288. Oil.

N'-Acetyl : needles from EtOH. M.p. 121-2°. N'-Benzoyl : needles from AcOH. M.p. 156°.

N : N'-Dibenzyl : cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 21°. B<sub>2</sub>HCl : needles from EtOH.Aq. + HCl. M.p. 149°.

N-Benzylidene : yellow cryst. from pet. ether. M.p. 60-1°. Sol. EtOH, Et<sub>2</sub>O, ligroin. N'-Acetyl : golden-yellow leaflets. M.p. 125°.

N : N'-Dibenzylidene : prisms from ligroin. M.p. 106°.

N-Acetyl : see o-Aminoacetanilide.

N : N'-Diacetyl : needles from H<sub>2</sub>O. M.p. 185-6°. Sol. boiling H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>, Me<sub>2</sub>CO, AcOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

N-Benzoyl : cryst. from H<sub>2</sub>O. M.p. 140°. Sol. EtOH.

N : N'-Dibenzoyl : prisms from AcOH. M.p. 301°. Spar. sol. most org. solvents. N-Formyl : needles from CHCl<sub>3</sub>-ligroin. M.p. 157°. Sol. Me<sub>2</sub>CO, AcOEt, hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O.

**N-Propionyl**: cryst. from  $\text{CHCl}_3$ -ligroin. M.p.  $124^\circ$ .

**N**: **N'-Di-o-nitrobenzoyl**: pale yellow needles from AcOH. M.p.  $265^\circ$ . Sol. hot EtOH.

**N**: **N'-Di-m-nitrobenzoyl**: needles from AcOH. M.p.  $240^\circ$ . Mod. sol. hot EtOH, AcOH. Spar. sol.  $\text{C}_6\text{H}_6$ .

**N-p-Nitrobenzoyl**: yellow needles from  $\text{H}_2\text{O}$ . M.p.  $200^\circ$ . Sol. EtOH, AcOH. Spar. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{C}_6\text{H}_6$ .

**N**: **N'-Di-p-nitrobenzoyl**: prisms from AcOH. M.p.  $267^\circ$ . Sol. hot EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

**N-Picryl**: red cryst. from xylene. M.p.  $177-8^\circ$  decomp. Sol.  $\text{Me}_2\text{CO}$ , xylene,  $\text{PhNO}_2$ . Spar. sol. EtOH,  $\text{Et}_2\text{O}$ .

**N-p-Toluenesulphonyl**: m.p.  $259-260.5^\circ$ .

Chapman, Perrott, *J. Chem. Soc.*, 1932, 1777.

Hinsberg, König, *Ber.*, 1895, **28**, 2947.

Jacobson, Lischke, *Ann.*, 1898, **303**, 378.

Fischer, Veiel, *Ber.*, 1905, **38**, 323.

Rupe, Porai-Koschitz, *Chem. Zentr.*, 1904, I, 102.

Walther, v. Pulawski, *J. prakt. Chem.*, 1899, **59**, 250.

Mixter, *Am. Chem. J.*, 1884, **6**, 27.

Wolff, *Ann.*, 1913, **399**, 302.

Pinnow, *Ber.*, 1899, **32**, 1402.

**m-Phenylenediamine** (1 : 3 - Diamino-benzene).

Rhombic cryst. from EtOH. M.p.  $63-4^\circ$ . B.p.  $282-4^\circ/760$  mm.  $D_{10}^{20}$  1.1421,  $D_4^{27.7}$  1.10696.  $n_D^{27.7}$  1.63390. Very sol.  $\text{H}_2\text{O}$ , EtOH. Less sol.  $\text{Et}_2\text{O}$ .

**N-Me**: see *N*-Methyl-*m*-phenylenediamine.

**N-Di-Me**: see *m*-Aminodimethylaniline.

**N**: **N'-Di-Me**: see *sym.*-Dimethyl-*m*-phenylenediamine.

**N-Et**: see *N*-Ethyl-*m*-phenylenediamine.

**N-Di-Et**: see *m*-Aminodiethylaniline.

**N-Phenyl**: see *m*-Aminodiphenylamine.

**N-Diphenyl**: see *m*-Aminotriphenylamine.

**N**: **N'-Diphenyl**: see *sym.*-Diphenyl-*m*-phenylenediamine.

**N**: **N'-Tri-Me**:  $\text{C}_9\text{H}_{14}\text{N}_2$ . MW, 150. B.p.  $270^\circ$  ( $280^\circ$ ). Sol. EtOH,  $\text{C}_6\text{H}_6$ . *Methiodide*: cryst. +  $\text{H}_2\text{O}$ . M.p.  $192^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ . *Acetyl*: cryst. from EtOH. M.p.  $68^\circ$ . B.p.  $280^\circ$ .

**N**: **N'-Tetra-Me**:  $\text{C}_{10}\text{H}_{16}\text{N}_2$ . MW, 164. Oil. F.p.  $-2^\circ$ . B.p.  $266-7^\circ/748$  mm.  $D_{10}^{20}$  0.9849. *Methiodide*: cryst. +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $192^\circ$ .

**N**: **N'-Tetraphenyl**:  $\text{C}_{30}\text{H}_{24}\text{N}_2$ . MW, 412. Needles from  $\text{Me}_2\text{CO}$ . M.p.  $137.5-138^\circ$ . Sol.

$\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Less sol. AcOH. Sol. 800 parts MeOH at  $15^\circ$ .

**N**: **N'-Di-p-tolyl**: see *Di-p-tolyl-m*-phenylenediamine.

**N-1-Naphthyl**: *m*-aminophenyl-1-naphthylamine.  $\text{C}_{16}\text{H}_{14}\text{N}_2$ . MW, 234. Prisms. M.p.  $94-5^\circ$ . B.p.  $275-80^\circ/12$  mm. Very sol. EtOH,  $\text{C}_6\text{H}_6$ . Sol. hot  $\text{Et}_2\text{O}$ . Mod. sol. hot  $\text{H}_2\text{O}$ .

**N-2-Naphthyl**: *m*-aminophenyl-2-naphthylamine.  $\text{C}_{16}\text{H}_{14}\text{N}_2$ . MW, 234. Needles from EtOH. M.p.  $128^\circ$ . B.p.  $320^\circ/40$  mm. Very sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . *B,2HCl*: m.p.  $210^\circ$ . *Picrate*: leaflets. M.p.  $180^\circ$  decomp. **N'-Acetyl**: needles from EtOH. M.p.  $135^\circ$ . **N**: **N'-Diacetyl**: m.p.  $147-8^\circ$ . **N'-Benzoyl**: m.p.  $173^\circ$ . **N**: **N'-Dibenzoyl**: m.p.  $213^\circ$ .

**Dinaphthyl**: see *Dinaphthyl-m*-phenylenediamine.

**N-Dibenzyl**:  $\text{C}_{20}\text{H}_{20}\text{N}_2$ . MW, 288. Oil. **N'-Acetyl**: needles from EtOH. M.p.  $143-4^\circ$ .

**N'-Benzoyl**: needles from AcOH. M.p.  $171-2^\circ$ .

**N**: **N'-Tetrabenzyl**: amorph. M.p.  $80-1^\circ$ .

**Dibenzylidene**: yellowish needles from  $\text{Et}_2\text{O}$ . M.p.  $104-5^\circ$ .

**N**: **N'-Diformyl**: cryst. from EtOH. M.p.  $155^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH.

**N-Acetyl**: see *m*-Aminoacetanilide.

**N**: **N'-Diacetyl**: prisms from EtOH.Aq. M.p.  $191^\circ$ .

**N-Benzoyl**: cryst. M.p.  $125^\circ$ .

**N**: **N'-Dibenzoyl**: needles from AcOH. M.p.  $240^\circ$ . Mod. sol. AcOH. Spar. sol. EtOH.

**N-m-Nitrobenzoyl**: m.p.  $142^\circ$ .

**N-p-Nitrobenzoyl**: light brown needles. M.p.  $212^\circ$ .

**N-Picryl**: orange-red cryst. from  $\text{Me}_2\text{CO}$ . M.p.  $206-7^\circ$ . Sol.  $\text{Me}_2\text{CO}$ . Spar. sol. EtOH, AcOH.

Zincke, Sintenis, *Ber.*, 1872, **5**, 792.

Jaubert, *Bull. soc. chim.*, 1899, **21**, 20.

Pinnow, Wegner, *Ber.*, 1897, **30**, 3110.

Gaess, Elsaesser, *Ber.*, 1893, **26**, 976.

Piccard, Brewster, *J. Am. Chem. Soc.*, 1921, **43**, 2630.

Desai, *J. Indian Chem. Soc.*, 1925, **5**, 425.

B.D.C., E.P., 168,689, (*Chem. Zentr.*, 1922, IV, 376; *Chem. Abstracts*, 1922, **16**, 720).

**p-Phenylenediamine** (1 : 4 - Diaminobenzene).

Plates from  $\text{Et}_2\text{O}$ . M.p.  $147^\circ$  ( $140^\circ$ ). B.p.  $267^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Sol. 100 parts cold  $\text{H}_2\text{O}$ . Heat of comb.  $\text{C}$ , 843.3 Cal.

**N-Me**: see *N*-Methyl-*p*-phenylenediamine.

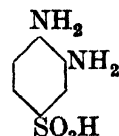
**N-Di-Me**: see *p*-Aminodimethylaniline.

**N**: **N'-Di-Me**: see *sym.*-Dimethyl-*p*-phenylenediamine.

N-Et : see N-Ethyl-p-phenylenediamine.  
 N-Di-Et : see p-Aminodiethylaniline.  
 N-Phenyl : see p-Aminodiphenylamine.  
 N-Diphenyl : see p-Aminotriphenylamine.  
 N : N'-Diphenyl : see sym.-Diphenyl-p-phenylenediamine.  
 N : N'-Tri-Me :  $C_9H_{14}N_2$ . MW, 150. Oil. B.p. 265°. Spar. sol.  $H_2O$ . Acetyl : m.p. 95°.  
 N : N'-Tetra-Me :  $C_{10}H_{16}N_2$ . MW, 164. Leaflets from EtOH.Aq. or ligroin. M.p. 51°. B.p. 260°. Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Sol. ligroin. Spar. sol. cold  $H_2O$ . Methiodide : leaflets. M.p. 265°.  
 N : N'-Tetra-Et :  $C_{14}H_{24}N_2$ . MW, 220. Plates from EtOH.Aq. M.p. 53°. B.p. 280°. Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , ligroin.  
 N-Propyl :  $C_9H_{14}N_2$ . MW, 150. Leaflets. B.p. 281°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .  
 N : N'-Dipropyl :  $C_{12}H_{20}N_2$ . MW, 192. Pale yellow oil. B.p. 155.5-156.5°/6 mm. Sol. EtOH. Spar. sol. hot  $H_2O$ . N-Chloroacetyl : prisms from EtOH. M.p. 121-121.5°.  
 N-Butyl : plates from pet. ether. M.p. 31.5°. B.p. 302.5-303.5°/760 mm. Sol. most org. solvents. Spar. sol. pet. ether. Insol.  $H_2O$ . Turns red in air.  $B_2HCl$  : plates from EtOH. Does not melt below 200°.  
 N-Isobutyl : leaflets from  $C_6H_6$ -ligroin. M.p. 39°.  
 N-Isoamyl : cryst. M.p. 31-2°.  
 N : N'-Triphenyl : cryst. from MeOH. M.p. 77-81°. B.p. 205-15°/0.01 mm. Very sol.  $Me_2CO$ , AcOEt,  $CS_2$ ,  $C_6H_6$ . N-Acetyl : leaflets from EtOH. M.p. 184°.  
 N : N'-Tetraphenyl : leaflets from  $Me_2CO$ . M.p. 199-200°. Very sol.  $C_6H_6$ . Spar. sol.  $Et_2O$ , EtOH, pet. ether. Sol. 90 parts boiling  $Me_2CO$ .  
 N-o-Tolyl : see 4'-Amino-2-methyldiphenylamine.  
 N-p-Tolyl : see 4'-Amino-4-methyldiphenylamine.  
 N : N'-Ditolyl : see Ditolyl-p-phenylenediamine.  
 N-Benzyl : plates from  $Et_2O$ . M.p. 37°. N'-Acetyl : needles from EtOH. M.p. 141-2°. N : N'-Diacetyl : prisms from EtOH.Aq. M.p. 116-17°. N'-Benzoyl : plates from EtOH. M.p. 182-3°. N : N'-Dibenzoyl : needles. M.p. 124°. N : N'-Dibenzyl : needles from EtOH. M.p. 89-90°. Sol.  $Et_2O$ . Spar. sol. EtOH- $FeCl_3$  → intense red col.  
 N : N'-Tetrabenzyl : needles from AcOH. M.p. 152° (149°). Sol.  $C_6H_6$ ,  $CHCl_3$ ,  $CS_2$ . Spar. sol. EtOH.  
 N-1-Naphthyl : plates. M.p. 80.5-81°. B.p. 275-80°/12 mm. Sol. hot EtOH,  $C_6H_6$ . Spar.

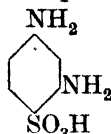
sol.  $H_2O$ . N'-Acetyl : needles from EtOH. M.p. 162-5°.  
 N-2-Naphthyl : needles from ligroin. M.p. 94°. Very sol.  $C_6H_6$ , EtOH,  $Et_2O$ ,  $Me_2CO$  with blue fluor.  $B_2HCl$  : m.p. 240°. decomp. N'-Acetyl : needles from  $C_6H_6$ . M.p. 160°.  
 Dinaphthyl : see Dinaphthyl-p-phenylenediamine.  
 N-Picryl : black prisms from AcOEt. M.p. 186-7°. Sol. AcOH, amyl alcohol.  
 Dibenzyldiene : plates from EtOH. M.p. 140°. N-Formyl : brownish needles from  $H_2O$ . M.p. 125-7°. N : N'-Di-formyl : m.p. 205-7°. N-Acetyl : see p-Aminoacetanilide.  
 N : N'-Diacetyl : leaflets from hot AcOH. M.p. 303°. N-Benzoyl : leaflets. M.p. 128°. Sol. EtOH,  $CHCl_3$ . Spar. sol.  $H_2O$ . N : N'-Dibenzoyl : leaflets. Does not melt below 300°. Spar. sol. EtOH,  $Et_2O$ , AcOH. N-m-Nitrobenzoyl : golden cryst. from EtOH. M.p. 217-18°. N-p-Nitrobenzoyl : cryst. M.p. 228°.  
 Paul, Z. angew. Chem., 1897, 10, 149.  
 Pomeranz, D.R.P., 269,542, (Chem. Zentr., 1914, I, 591).  
 Wurster, Ber., 1879, 12, 523.  
 Reilly, Hickinbottom, J. Chem. Soc., 1917, 111, 1032.  
 Ullmann, Dahmen, Ber., 1908, 41, 3750.  
 Philip, J. prakt. Chem., 1886, 34, 65.  
 Jacobs, Heidelberg, J. Biol. Chem., 1915, 21, 115.  
 Quick, J. Am. Chem. Soc., 1920, 42, 1033.  
 Norris, Cummings, J. Ind. Eng. Chem., 1925, 17, 305.  
 Major, J. Am. Chem. Soc., 1931, 53, 4375.  
 Andrews, Lowy, J. Am. Chem. Soc., 1934, 56, 1411.  
 Hofmann, Compt. rend., 1863, 56, 994.

**o-Phenylenediamine-4-sulphonic Acid**  
 (3 : 4-Diaminobenzenesulphonic acid)



$C_6H_5O_3N_2S$  MW, 188  
 Needles. Decomp. on melting. Mod. sol. hot  $H_2O$ . Turns blue or green in air.  $FeCl_3$  → reddish-brown col.  
 Nietzki, Lerch, Ber., 1888, 21, 3221.  
 Post, Hartung, Ann., 1880, 205, 100.

**m-Phenylenediamine-4-sulphonic Acid**  
(2 : 4-Diaminobenzenesulphonic acid)



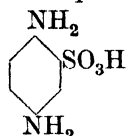
$C_6H_8O_3N_2S$  MW, 188

Plates and prisms from  $H_2O$ . Spar. sol. cold  $H_2O$ . Slowly turns brown in air.

Post, Hartung, *Ann.*, 1880, 205, 107.

Hunter, Sprung, *J. Am. Chem. Soc.*, 1931, 53, 1440.

**p-Phenylenediamine-2-sulphonic Acid**  
(2 : 5-Diaminobenzenesulphonic acid)



$C_6H_8O_3N_2S$  MW, 188

Plates +  $2H_2O$  from  $H_2O$ , needles from  $H_2O$  with trace  $H_2SO_4$ . Mod. sol.  $H_2O$ . Insol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

N-Di-Me: sol.  $H_2O$ .  $FeCl_3 \rightarrow$  red col.

Anilide: leaflets from  $H_2O$ . M.p. 171°.

B, HCl: needles. M.p. 215° decomp.

A.G.F.A., D.R.P., 204,972, (*Chem. Zentr.*, 1909, I, 475).

Kalle, D.R.P., 124,907, (*Chem. Zentr.*, 1901, II, 1103).

Fischer, *Ber.*, 1891, 24, 3789.

Eger, *Ber.*, 1889, 22, 848.

**Phenylene Dimercaptan.**

See Dithiocatechol, Dithiohydroquinone, and Dithioresorcinol.

**Phenylenedimethyldiamine.**

See sym.-Dimethylphenylenediamine.

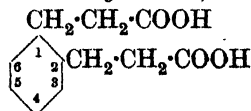
**Phenylenedinaphthyldiamine.**

See Dinaphthylphenylenediamine.

**Phenylenediphenyldiamine.**

See sym.-Diphenylphenylenediamine.

**o-Phenylenedipropionic Acid** (o-Xylylene-diacetic acid, benzene-o-dipropionic acid, o-diethylbenzene- $\omega$  :  $\omega'$ -dicarboxylic acid)



$C_{12}H_{14}O_4$  MW, 222

Needles from  $H_2O$ , prisms from EtOH.Aq. M.p. 171° (168°, 162°).

Di-Et ester:  $C_{16}H_{22}O_4$ . MW, 278. B.p. 200-2°/12 mm.

Diamide:  $C_{12}H_{16}O_2N_2$ . MW, 220. Needles from EtOH. M.p. 195°.

Titely, *J. Chem. Soc.* 1928, 2578.

Fries, Bestian, *Ber.*, 1936, 69, 720.

**m-Phenylenedipropionic Acid** (m-Xylylene-diacetic acid, benzene-m-dipropionic acid, m-diethylbenzene- $\omega$  :  $\omega'$ -dicarboxylic acid).

Plates from  $H_2O$ . M.p. 146-7°. Mod. sol. EtOH,  $Et_2O$ .

Di-Me ester:  $C_{14}H_{18}O_4$ . MW, 250. Leaflets from MeOH.Aq. M.p. 51°. Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

Di-Et ester: liq. B.p. 247-50°/60 mm.

Kipping, *J. Chem. Soc.*, 1888, 53, 33.

**p-Phenylenedipropionic Acid** (p-Xylylene-diacetic acid, benzene-p-dipropionic acid, p-diethylbenzene- $\omega$  :  $\omega'$ -dicarboxylic acid).

Cryst. from MeOH. M.p. 223-4°. Spar. sol. EtOH. Insol.  $H_2O$ .

Di-Me ester: plates from MeOH. M.p. 115°. Spar. sol. cold MeOH.

See previous reference.

**Phenyleneditolyldiamine.**

See Ditolylphenylenediamine.

**Phenylerythrene.**

See Phenyl-1 : 3-butadiene.

**Phenylethane.**

See Ethylbenzene.

**Phenyl p-ethoxyphenacyl Ether.**

See under p-Hydroxyphenacyl Alcohol.

**Phenyl ethoxystyryl Ketone.**

See under Hydroxychalkone.

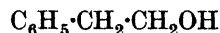
**$\beta$ -Phenylethylacetylene.**

See 4-Phenylbutine-1.

**1-Phenylethyl Alcohol.**

See Methylphenylcarbinol.

**2-Phenylethyl Alcohol** (Benzylcarbinol,  $\beta$ -hydroxyethylbenzene)



$C_8H_{10}O$  MW, 122

Found in fresh rose petals and neroli oil. B.p. 219-21°/750 mm., 104°/14 mm., 98-100°/12 mm.  $D_4^{25}$  1.0235.  $n_D^{18}$  1.5337. Mod. sol.  $H_2O$ , EtOH.Aq. Used in perfumery, as also are its esters.

Me ether: see Methyl phenylethyl Ether.

Phenyl ether: phenyl phenylethyl ether.

$C_{14}H_{14}O$ . MW, 198. B.p. 166°/14 mm.

Formyl: b.p. 94°/9 mm.  $D^{18}$  1.054.

Acetyl: b.p. 224°, 118-20°/13 mm.  $D^{18}$  1.038.  $n_D$  1.5108.

Methoxyacetyl: b.p. 149°/18 mm.  $D_4^{17}$  1.0806.  $n_D^{17}$  1.5000.

*Oxalate*: cryst. from 80% EtOH. M.p. 51·5°.

*p*-Nitrobenzoyl: m.p. 62–3°.

*Allophanate*: m.p. 186°.

*Phenylurethane*: m.p. 79–80°.

Skita, Ritter, *Ber.*, 1911, **43**, 3398.

Bouveault, Blanc, *Bull. soc. chim.*, 1904, **31**, 674.

Skita, *Ber.*, 1915, **48**, 1694.

Skita, Mayer, *Ber.*, 1912, **45**, 3584.

Leonard, *J. Am. Chem. Soc.*, 1925, **47**, 1774.

### Phenylethylamine.

*See* Aminoethylbenzene.

### $\beta$ -Phenylethylbenzylamine.

*See* Benzyl-phenylethylamine.

### Phenylethyl bromide.

*See* Bromoethylbenzene.

### Phenylethyl chloride.

*See* Chloroethylbenzene.

### Phenylethylene.

*See* Styrene.

### Phenylethylene bromohydrin.

*See* Styrene bromohydrin.

### Phenylethylene chlorohydrin.

*See* Styrene chlorohydrin.

### Phenylethylenediamine ( $\alpha\beta$ -Diaminoethylbenzene)

$C_6H_5 \cdot CH(NH_2) \cdot CH_2NH_2$   
 $C_8H_{12}N_2$  MW, 136

*l*-.  
 M.p. 4–5°. B.p. 104°/1–2 mm.  $D^{18}_D$  1·034.  
 $[\alpha]_D^{20} - 35·2^\circ$ .

*N*:*N'*-Diacetyl: cryst. from AcOEt. M.p. 174°.

*N*:*N'*-Dibenzoyl: cryst. from EtOH. M.p.

227°.  
*d*-Tartrate: m.p. 220° decomp.  $[\alpha]_D + 40·8^\circ$  in  $H_2O$ .

*dl*-.  
 B.p. 243–6°, 156–7°/42 mm. Very sol.  $H_2O$ .  
 Sol. most org. solvents.

*Diacetyl*: cryst. from AcOEt. M.p. 159° (152°).

*Triacetyl*: plates from  $H_2O$ . M.p. 166°.

*Dibenzoyl*: leaflets from EtOH. M.p. 217°.

*Picrate*: yellow cryst. M.p. 160°. Insol.  $C_6H_6$ , ligroin.

*B*,*2HCl*: needles. Does not melt below 260°.

Reihlen, Hessling, Hühn, Weinbrenner, *Ann.*, 1932, **493**, 25.

Kanewskaja, *J. prakt. Chem.*, 1932, **132**, 340.

Reihlen, Weinbrenner, Hessling, *Ann.*, 1932, **494**, 157.

Feist, Arnstein, *Ber.*, 1893, **28**, 425, 3172.

### Phenylethylene dibromide.

*See* Styrene dibromide.

### Phenylethylene dichloride.

*See* Styrene dichloride.

### Phenylethylene Glycol.

*See* Styrene Glycol.

### Phenylethylene oxide.

*See* Styrene oxide.

### Phenylethylhydrazine (*Phenethylhydrazine*, $\omega$ -hydrazinoethylbenzene)

$C_6H_5 \cdot CH_2 \cdot CH_2 \cdot NH \cdot NH_2$   
 $C_8H_{12}N_2$  MW, 136

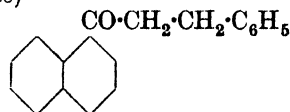
B.p. 137–9°/12–13 mm.

*B*,*HCl*: plates from EtOH. M.p. 171°.

*Dibenzoyl*: needles from 50% EtOH. M.p. 144–5°.

Votoček, Lemings, *Chem. Zentr.*, 1932, **II**, 1613.

### Phenylethyl 1-naphthyl Ketone ( $\omega$ -Benzyl- $\alpha$ -acetonnaphthone)



$C_{19}H_{16}O$  MW, 260

Leaflets from 70% EtOH. M.p. 93° (53–4°).  
 B.p. 240–60°/25 mm. Sol. conc.  $H_2SO_4$  with yellow col. turning brownish-red on warming.

*Oxime*: cryst. from EtOH. M.p. 120°.

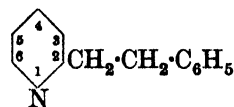
Albrecht, *Monatsh.*, 1914, **35**, 1498.

Bergmann, Weiss, *Ber.*, 1931, **64**, 1492.

### Phenylethylpiperidine.

*See* Stilbazoline.

### 2-Phenylethylpyridine (*Dihydro- $\alpha$ -stilbazole*, *phenyl-2-pyridylethane*)



$C_{13}H_{13}N$  MW, 183

Liq. with sweet odour. F.p. –3°. B.p. 289·5°/766 mm.  $D^{20}_D$  1·0465. Very sol. EtOH, Et<sub>2</sub>O,  $C_6H_6$ . Spar. sol.  $H_2O$ . Volatile in steam.

*B*,*H**AuCl*<sub>4</sub>: yellow needles from dil. HCl. M.p. 149–50°.

*B*,*HCl*, *HgCl*<sub>2</sub>: cryst. M.p. 149°.

*B*<sub>2</sub>, *H*<sub>2</sub>, *PtCl*<sub>6</sub>: reddish-yellow needles. M.p. 185–6°.

Baurath, *Ber.*, 1888, **21**, 821.

### 4-Phenylethylpyridine ( $\gamma$ -*Dihydrostilbazole*, *phenyl-4-pyridylethane*).

Needles from EtOH. M.p. 69–71° (65°).

$B, HCl$ : cryst. from  $EtOH-Et_2O$ . M.p.  $180^\circ$ .  
 $B, HI$ : brownish-red leaflets from  $H_2O$ . De-comp. about  $150^\circ$ .

$B, HAuCl_4$ : yellow leaflets. M.p.  $183-5^\circ$  ( $166^\circ$ ).

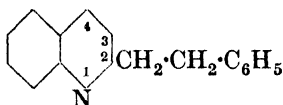
$B, H_2PtCl_6$ : brown leaflets. M.p.  $214^\circ$ .

*Picrate*: yellow needles. M.p.  $162-3^\circ$ .

Friedländer, *Ber.*, 1905, **38**, 2837.

Fels, *Ber.*, 1904, **37**, 2147.

**2-Phenylethylquinoline** (*Phenyl-2-quinolyethane*)



$C_{17}H_{15}N$

MW, 233

Cryst. from  $C_6H_6$  or ligroin. M.p.  $28^\circ$ . B.p.  $214-19^\circ/14$  mm. Spar. volatile in steam.

*Picrate*: pale yellow prisms. M.p. about  $130^\circ$ .

Heymann, Koenigs, *Ber.*, 1888, **21**, 1426.

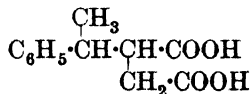
Ziegler, Zeiser, *Ann.*, 1931, **485**, 190.

**4-Phenylethylquinoline** (*Phenyl-4-quinolyethane*).

Cryst. from  $EtOH.Aq.$  or ligroin. M.p.  $100-1^\circ$ .

See first reference above.

**$\alpha$ -Phenylethyl-succinic Acid** (*3-Phenylbutane-1:2-dicarboxylic acid*)



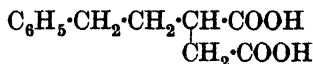
$C_{12}H_{14}O_4$

MW, 222

Needles from  $C_6H_6$  or hot  $H_2O$ . M.p.  $144-146.5^\circ$ . Sol.  $EtOH, Et_2O$ . Spar. sol. cold  $C_6H_6$ .

Stobbe, *Ann.*, 1899, **308**, 127.

**$\beta$ -Phenylethyl-succinic Acid** (*4-Phenylbutane-1:2-dicarboxylic acid*)



$C_{12}H_{14}O_4$

MW, 222

Cryst. from  $H_2O$  or  $C_6H_6$ . M.p.  $136^\circ$ . Sol.  $EtOH, Et_2O$ . Spar. sol.  $C_6H_6$ . Insol.  $CS_2$ , low-boiling pet. ether.

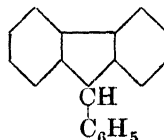
*Mono-NH<sub>4</sub> salt*: leaflets from  $H_2O$ . M.p.  $185^\circ$  decomp.

*Anhydride*:  $C_{12}H_{12}O_3$ . MW, 204. Cryst. from pet. ether. M.p.  $56^\circ$ . Sol.  $CHCl_3, C_6H_6$ . Spar. sol.  $CS_2$ , pet. ether.

Thiele, Meisenheimer, *Ann.*, 1899, **306**, 257, 261.

**9-Phenylfluorene**

(*Phenyl-diphenylene-methane*)



$C_{19}H_{14}$

MW, 242

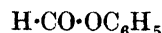
Needles or leaflets from  $EtOH$  or  $C_6H_6$ . M.p.  $148^\circ$  ( $145^\circ$ ). Sol.  $AcOH, CHCl_3, C_6H_6$ , pet. ether, hot  $EtOH$ . Spar. sol.  $Et_2O$ . Alc. and benzene solutions show blue fluor.  $CrO_3 \rightarrow$  o-benzoylbenzoic acid.

Vorländer, Pritzsche, *Ber.*, 1913, **46**, 1796.

Kliegl, *Ber.*, 1905, **38**, 287.

Koelsch, *J. Am. Chem. Soc.*, 1934, **56**, 482.

**Phenyl formate**



$C_7H_6O_2$

MW, 122

B.p.  $173^\circ$  slight decomp.,  $107^\circ/25$  mm.  $D^{20}_{400}$  1.0879.

Seifert, *J. prakt. Chem.*, 1885, **31**, 467.

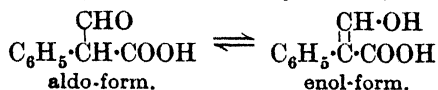
Auger, *Compt. rend.*, 1904, **139**, 799.

Adickes, Brunnert, Lückner, *J. prakt. Chem.*, 1931, **130**, 174.

**Phenylformylacetanilide.**

See under Phenylformylacetic Acid.

**Phenylformylacetic Acid** (*Phenylaldehyde-acetic acid,  $\beta$ -hydroxyatropic acid,  $\alpha$ -hydroxymethylenephénylacetic acid, 2-hydroxy-1-phenylacrylic acid, phenylmalonaldehydic acid*)



$C_9H_8O_3$

MW, 164

The acid is unknown in the free state.

*Me ester*:  $C_{10}H_{10}O_3$ . MW, 178.  $\alpha$ (*enol*)-form: leaflets. M.p.  $40-1^\circ$ .  $\beta$ (probably *aldo*)-form: leaflets from  $C_6H_6$ . M.p.  $91-3^\circ$  ( $87^\circ$ ). *Semicarbazone*: m.p.  $159-60^\circ$ . *Aldo-enol* mixture: b.p.  $153-5^\circ/18$  mm.  $n^{20}_D$  1.52425.

*Et ester*:  $C_{11}H_{12}O_3$ . MW, 192.  $\alpha$ (mainly *enol*)-form: b.p.  $135^\circ/15$  mm. Alc. sol.  $\rightarrow$  bluish-violet col. with  $FeCl_3$ . *Formyl*: m.p.  $87^\circ$ . *Acetyl*: b.p.  $184^\circ/18$  mm.  $\beta$ (probably *aldo*)-form: m.p.  $50^\circ$  ( $70^\circ$ ). *Semicarbazone*: m.p.  $162-5^\circ$  ( $130-1^\circ$ ). *Aldo-enol* mixture: b.p.  $150-1^\circ/18$  mm.  $n^{20}_D$  1.532.  $\gamma$ -form: plates. M.p.  $108-10^\circ$ .

*Nitrile*:  $\alpha$ -formylbenzyl cyanide,  $\alpha$ -cyano-phenylacetaldehyde.  $C_9H_7ON$ . MW, 145. Leaf-



lets from EtOH. M.p. 165–6° (157–8°). Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, hot H<sub>2</sub>O.

*Anilide*: phenylformylacetanilide. Exists in two modifications. (α-) Plates from pet. ether. M.p. 68°. (β-) Triangular plates from Et<sub>2</sub>O or AcOH. M.p. 98°.

*Piperidide*: exists in two modifications. (α-) M.p. 104–6°. (β-) M.p. 122°.

Décombe, *Ann. chim.*, 1932, **18**, 90, 113.

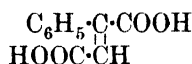
Wislicenus, *Erbe, Ann.*, 1920, **421**, 119.

Tiffeneau, *Levy, Chem. Abstracts*, 1930, **24**, 2450.

Ghosh, *J. Chem. Soc.*, 1916, **109**, 113.

Wislicenus, *Ann.*, 1917, **413**, 206.

### Phenylfumaric Acid



C<sub>10</sub>H<sub>8</sub>O<sub>4</sub> MW, 192

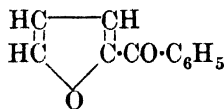
Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 128–9°. Very sol. H<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Above m.p. → phenylmaleic anhydride. Reduces KMnO<sub>4</sub>.Aq. slowly.

Almström, *Ber.*, 1915, **48**, 2009; *Ann.*, 1916, **411**, 375.

### Phenyl furyl Diketone.

See Benzfuryl.

**Phenyl α-furyl Ketone** (2-Benzoylfuran, α-benzofuran)



C<sub>11</sub>H<sub>8</sub>O<sub>2</sub> MW, 172

B.p. 285°, 186°/46 mm., 164°/19 mm. D<sub>20</sub><sup>20</sup> 1.1732. n<sub>D</sub><sup>20</sup> 1.6055 (1.5798).

*Oxime*: yellow needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 138° (132°). Very sol. org. solvents.

*Semicarbazone*: m.p. 182°.

Asahina, *Murayama, Arch. Pharm.*, 1914, **252**, 448.

Marquis, *Bull. soc. chim.*, 1900, **23**, 33.

Gilman, *Hewlett, Chem. Abstracts*, 1930, **24**, 1640.

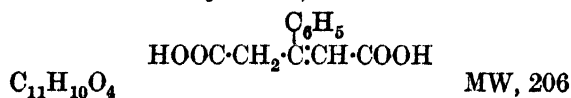
### Phenyl furylvinyl Ketone.

See Furfurylideneacetophenone.

### Phenyl-gamma Acid.

See N-Phenyl-2-amino-8-naphthol-6-sulphonic Acid.

**2-Phenylglutaconic Acid** (2-Phenylpropylene-1 : 3-dicarboxylic acid)



Prismatic plates from AcOEt. M.p. 154–5°. Above m.p. → anhydride. P + HI at 150° → 2-phenylglutaric acid.

*Mono-Et ester*: C<sub>13</sub>H<sub>14</sub>O<sub>4</sub>. MW, 234. Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 78°. Dist. → anhydride.

*Di-Et ester*: C<sub>15</sub>H<sub>16</sub>O<sub>4</sub>. MW, 262. B.p. 186–7°/11 mm., 167–8°/5 mm. D<sub>4</sub><sup>20</sup> 1.1014. n<sub>D</sub><sup>20</sup> 1.5240.

*Monoamide*: C<sub>11</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 205. Needles from AcOEt. M.p. 138°.

*Mononitrile*: C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 187. Leaflets from EtOH or AcOH. M.p. 256–7°. Spar. sol. H<sub>2</sub>O, EtOH.

*Anhydride*: C<sub>11</sub>H<sub>8</sub>O<sub>3</sub>. MW, 188. Leaflets from AcOEt. M.p. 206°.

*Imide*: C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 187. Prisms from AcOH, leaflets from EtOH. M.p. 256–7°. Spar. sol. boiling EtOH. Very spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O. Alc. FeCl<sub>3</sub> → red col.

*Monoanilide*: cryst. from AcOEt. M.p. 174°.

*Mono-p-toluidide*: cryst. from AcOEt. M.p. 184°.

Michael, *J. prakt. Chem.*, 1894, **49**, 22.

Ruhemann, *J. Chem. Soc.*, 1899, **75**, 252.

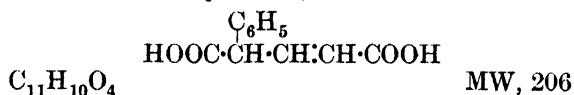
Feist, *Pomme, Ann.*, 1909, **370**, 74.

Guareschi, *Chem. Zentr.*, 1907, **I**, 459; 1901, **I**, 821.

Bland, *Thorpe, J. Chem. Soc.*, 1912, **101**, 868.

Gidvani, *Kon, J. Chem. Soc.*, 1932, 2448.

**3-Phenylglutaconic Acid** (3-Phenylpropylene-1 : 3-dicarboxylic acid)

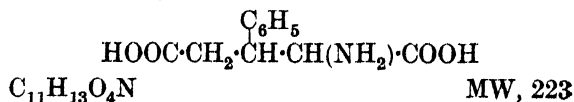


Cryst. from EtOH. M.p. 164°.

*Di-Et ester*: b.p. 220°/80 mm. D<sub>20</sub><sup>20</sup> 1.0712. n<sub>D</sub><sup>20</sup> 1.50923.

Phalnikar, *Nargund, Chem. Zentr.*, 1936, **I**, 4556.

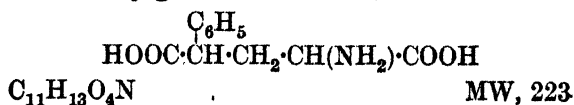
### 2-Phenylglutamic Acid



Plates from H<sub>2</sub>O. M.p. 179°. Insol. EtOH. N-Benzoyl: needles from H<sub>2</sub>O. M.p. 171–2°.

Harington, *J. Biol. Chem.*, 1925, **64**, 29.

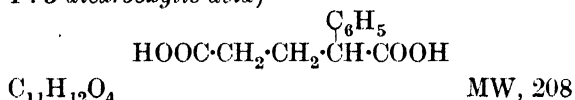
### 3-Phenylglutamic Acid



Rhombic cryst. M.p. 185° decomp. Spar. sol. H<sub>2</sub>O.

v. Beznák, *Biochem. Z.*, 1929, **205**, 414.

**1-Phenylglutaric Acid** (1-Phenylpropane-1 : 3-dicarboxylic acid)

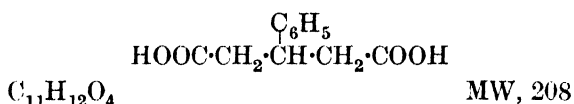


Cryst. from C<sub>6</sub>H<sub>6</sub> or Et<sub>2</sub>O-pet. ether. M.p. 82-3°. Dist. in vacuo → anhydride.

Anhydride: C<sub>11</sub>H<sub>10</sub>O<sub>3</sub>. MW, 190. Needles from Et<sub>2</sub>O. M.p. 95°. B.p. 218-30°/13 mm.

Fichter, Merckens, *Ber.*, 1901, **34**, 4175.

**2-Phenylglutaric Acid** (Benzylidenediacetic acid, 2-phenylpropane-1 : 3-dicarboxylic acid)



Plates from H<sub>2</sub>O, prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 140°. Sol. EtOH, Et<sub>2</sub>O, AcOEt, CHCl<sub>3</sub>. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. pet. ether.  $k = 7.7 \times 10^{-5}$  at 25°. Above m.p. → anhydride.

Di-Me ester: C<sub>13</sub>H<sub>16</sub>O<sub>4</sub>. MW, 236. Prisms or needles from MeOH. M.p. 86-7°.

Di-Et ester: C<sub>15</sub>H<sub>20</sub>O<sub>4</sub>. MW, 264. B.p. 188-9°/13 mm.

Dichloride: C<sub>11</sub>H<sub>10</sub>O<sub>2</sub>Cl<sub>2</sub>. MW, 245. M.p. 46°. B.p. 178-80°/18 mm.

Anhydride: C<sub>11</sub>H<sub>10</sub>O<sub>3</sub>. MW, 190. Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 105°. B.p. 217-19°/15 mm. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. pet. ether.

Imide: C<sub>11</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 189. Leaflets from H<sub>2</sub>O, EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 173-4°.

Monoanilide: needles from EtOH.Aq. M.p. 171° (168°).

Mono-p-toluidide: needles. M.p. 154-5°.

Anil: needles from EtOH. M.p. 223°.

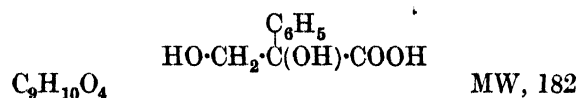
Michael, *J. prakt. Chem.*, 1887, **35**, 352.

Herrmann, Vorländer, *Chem. Zentr.*, 1899, **I**, 730.

Avery, Bouton, *Am. Chem. J.*, 1898, **20**, 513.

v. Braun, Weissbach, *Ber.*, 1931, **64**, 1787.

**1-Phenylglyceric Acid** (1 : 2-Dihydroxy-1-phenylpropionic acid, atroglyceric acid)



Cryst. from H<sub>2</sub>O. M.p. 146°. Mod. sol. hot H<sub>2</sub>O. Heat → phenylacetaldehyde.

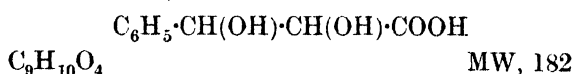
Nitrile: C<sub>9</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 163. Needles from Et<sub>2</sub>O. M.p. 55-7° decomp.

Plöchl, Blümlein, *Ber.*, 1883, **16**, 1292.

Fittig, Kast, *Ann.*, 1881, **206**, 30.

McKenzie, Wood, *J. Chem. Soc.*, 1919, **115**, 838.

**2-Phenylglyceric Acid** (αβ-Dihydroxyhydrocinnamic acid)



Exists in two stereoisomeric forms.

(i) d-.

Needles or plates from C<sub>6</sub>H<sub>6</sub>. M.p. 95°. Sol. 10 parts Et<sub>2</sub>O at 20°.  $[\alpha]_D^{20} + 26.1^\circ$  in H<sub>2</sub>O, + 21.15° in EtOH, + 27.5° in Me<sub>2</sub>CO.

l-.

Cryst. M.p. 97-8°.  $[\alpha]_D - 25.6^\circ$ .

dl-.

Needles from Et<sub>2</sub>O. M.p. 122°. Sol. 15 parts Et<sub>2</sub>O.  $k = 2.35 \times 10^{-4}$ .

Me ester: C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>. MW, 196. Cryst. M.p. 87°. B.p. 110°/0.1 mm. Dibenzoyl: cryst. from EtOH. M.p. 113.5°.

Monoacetyl: cryst. from H<sub>2</sub>O. M.p. 158°.

Dibenzoyl: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 187° decomp. Sol. EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, hot H<sub>2</sub>O.

Et ester: prisms from toluene. M.p. 109°.

Phenylhydrazide: m.p. 177°.

(ii) d-.

Plates from H<sub>2</sub>O. M.p. 166-7° (164°). Sol. 200 parts Et<sub>2</sub>O, 25 parts H<sub>2</sub>O at 20°.  $[\alpha]_D^{20} + 39.6^\circ$  in H<sub>2</sub>O.

l-.

Plates from H<sub>2</sub>O. M.p. 166-7° (164°). Sublimes in high vacuum.  $[\alpha]_D^{20} - 39.6^\circ$  in H<sub>2</sub>O, - 30.5° in EtOH, - 36.4° in Me<sub>2</sub>CO.

dl-.

Plates from H<sub>2</sub>O, leaflets from Et<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>. M.p. 141-2°. Very sol. H<sub>2</sub>O, EtOH. Mod. sol. Me<sub>2</sub>CO. Spar. sol. CHCl<sub>3</sub>, CS<sub>2</sub>. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin. Sol. 75 parts Et<sub>2</sub>O at 20°.  $k = 2.54 \times 10^{-4}$ . At 160° → phenylacetaldehyde.

Me ester: cryst. M.p. 67°. B.p. 114°/0.1 mm.

Et ester: dibenzoyl, needles from EtOH. M.p. 85°.

Monoacetyl: cryst. from H<sub>2</sub>O. M.p. 93.5°.

Diacetyl: leaflets +  $\frac{1}{2}$  H<sub>2</sub>O from H<sub>2</sub>O. M.p. 88-9°.

Amide: C<sub>9</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 181. Leaflets from EtOH. M.p. 161-2°. Sol. EtOH, Me<sub>2</sub>CO, hot H<sub>2</sub>O.

*Phenylhydrazide*: m.p. 215°.

Anschütz, Kinnicutt, *Ber.*, 1879, **12**, 539.

Plöchl, Mayer, *Ber.*, 1897, **30**, 1601.

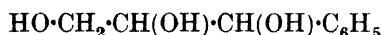
Lipp, *Ber.*, 1883, **16**, 1289.

Riiber, *Ber.*, 1908, **41**, 2413.

Riiber, Berner, *Ber.*, 1917, **50**, 894.

Berner, *Chem. Zentr.*, 1919, III, 777.

**1-Phenylglycerol** (*Stycerine*, 1:2:3-tri-hydroxy-1-phenylpropane)



$\text{C}_9\text{H}_{12}\text{O}_3$  MW, 168

Cryst. from EtOH. M.p. 100·5° (98-9°). B.p. 184-6°.  $D_4^{18.5}$  1·2213.  $n_D^{15}$  1·5600.

1-*Me ether*:  $\text{C}_{10}\text{H}_{14}\text{O}_3$ . MW, 182. M.p. 68°.

2-*Me ether*: m.p. 44°. B.p. 153-6°/5-6 mm.

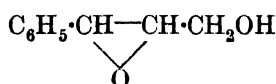
3-*Me ether*: needles. M.p. 54°. B.p. 159°/8 mm. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol. pet. ether.

Platt, Hibbert, *Chem. Zentr.*, 1933, I, 3923.

Tiffeneau, Neuberg-Rabinovitch, Cahmann, *Bull. soc. chim.*, 1935, **2**, 1869.

See also Prévost, Losson, *Compt. rend.*, 1934, **198**, 659.

**Phenylglycide** (*Phenylglycidol*)



$\text{C}_9\text{H}_{10}\text{O}_2$  MW, 150

Waxy cryst. M.p. 26·5°. B.p. 134-5°/4-5 mm.  $n_D^{27}$  1·5427.

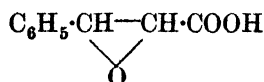
*Me ether*:  $\text{C}_{10}\text{H}_{12}\text{O}_2$ . MW, 164. B.p. 130-7°/21-3 mm.  $n_D^{30}$  1·5170.

*Acetyl*: b.p. 129-32°/3 mm.  $n_D^{22}$  1·5208.

*Phenylurethane*: cryst. from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 87°.

Jahn, Hibbert, *Chem. Zentr.*, 1933, II, 1018.

**Phenylglycidic Acid**



$\text{C}_9\text{H}_8\text{O}_3$  MW, 164

*l.*

*Na salt*:  $[\alpha]_D$  -157·9° in  $\text{H}_2\text{O}$ .

*dl.*

Prisms. M.p. 83-4°. B.p. 128-30°/4-5 mm. Spar. sol.  $\text{H}_2\text{O}$ . Stable at ord. temps. Above

m.p.  $\rightarrow$  phenylacetaldehyde. Dil.  $\text{H}_2\text{SO}_4$   $\rightarrow$  phenylacetaldehyde + 2-phenylglyceric acid (both forms).

*Et ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_3$ . MW, 192. B.p. 279·5° slight decomp.

*Anilide*: needles from EtOH. M.p. 142°.

Erlenmeyer, *Ber.*, 1906, **39**, 789.

Dieckmann, *Ber.*, 1910, **43**, 1035.

Erdmann, D.R.P., 107,228.

I.G., F.P., 715,657, (*Chem. Zentr.*, 1932, II, 2747).

Kaufmann, D.R.P., 515,034, (*Chem. Zentr.*, 1931, I, 1829).

**C-Phenylglycine.**

$\alpha$ -Aminophenylacetic Acid, *q.v.*

**Phenylglycine** (*Anilinoacetic acid*)



$\text{C}_8\text{H}_9\text{O}_2\text{N}$  MW, 151

Cryst. M.p. 127-8°. Mod. sol.  $\text{H}_2\text{O}$ . Less sol.  $\text{Et}_2\text{O}$ .  $k = 3\cdot8 \times 10^{-6}$  at 25°. Na and K salts sol.  $\text{H}_2\text{O}$ , Ca salt spar. sol.  $\text{H}_2\text{O}$ , Fe and Cu salts insol.  $\text{H}_2\text{O}$ . Intermediate in manufacture of indigo.

*Me ester*:  $\text{C}_9\text{H}_{11}\text{O}_2\text{N}$ . MW, 165. Needles. M.p. 48°. Sol. EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ . Volatile in steam.

*Et ester*:  $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$ . MW, 179. Leaflets. M.p. 58°. B.p. 273-4°, 163°/18 mm., 140°/8 mm. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. hot  $\text{H}_2\text{O}$ . Volatile in steam. *N-Carboethoxyl*: thick liq. B.p. 177-8°/14 mm.

*Isoamyl ester*:  $\text{C}_{13}\text{H}_{19}\text{O}_2\text{N}$ . MW, 221. Leaflets from  $\text{C}_6\text{H}_6$ . M.p. 37-9°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , amyl alcohol. Insol.  $\text{H}_2\text{O}$ . *N-Isoamyl*:  $\text{C}_{18}\text{H}_{29}\text{O}_2\text{N}$ . MW, 291. B.p. 215-16°/20 mm.

*Phenyl ester*:  $\text{C}_{14}\text{H}_{13}\text{O}_2\text{N}$ . MW, 227. Leaflets from cold EtOH.Aq. M.p. 82-3°. Sol. cold EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Hot EtOH  $\rightarrow$  phenylglycine ethyl ester.

*p-Tolyl ester*:  $\text{C}_{15}\text{H}_{15}\text{O}_2\text{N}$ . MW, 241. Pale yellow needles from pet. ether. M.p. 109°.

*Amide*:  $\text{C}_8\text{H}_{10}\text{ON}_2$ . MW, 150. Needles from  $\text{H}_2\text{O}$ , leaflets from EtOH. M.p. 136°. Sol. EtOH,  $\text{Me}_2\text{CO}$ , hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ .

*Methylamide*: m.p. 118°.

*Ethylamide*: m.p. 53-4°.

*Anilide*: needles from EtOH. M.p. 113°. Sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ .

*p-Toluidide*: needles. M.p. 171-2° (165°). Sol. EtOH,  $\text{Et}_2\text{O}$ .

*Nitrile*:  $\text{C}_8\text{H}_8\text{N}_2$ . MW, 132. Plates from ligroin- $\text{Et}_2\text{O}$ . M.p. 48°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin.

**Hydrazide**: leaflets from EtOH. M.p. 126-5°. Sol. warm H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O.

**Piperidide**: m.p. 102-3°.

**N-Me**: see *N*-Phenylsarcosine.

**N-Et**: C<sub>10</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 179. Oil. *Et ester*: C<sub>12</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 207. B.p. 280°/759 mm., 178°/42 mm. Turns brown in air. *Isoamyl ester*: C<sub>15</sub>H<sub>23</sub>O<sub>2</sub>N. MW, 249. B.p. 187°/18 mm. *Amide*: C<sub>10</sub>H<sub>14</sub>ON<sub>2</sub>. MW, 178. M.p. 114-15°. *Nitrile*: C<sub>10</sub>H<sub>12</sub>N<sub>2</sub>. MW, 160. Cryst. M.p. about 24°. B.p. 183°/20 mm., 150-1°/13 mm.

**N-Phenyl**: see *N*-Diphenylglycine.

**N-Formyl**: see Formylphenylglycine.

**N-Acetyl**: *N*-phenylaceturic acid. C<sub>10</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 193. Leaflets from H<sub>2</sub>O. M.p. 194°. Sol. EtOH, AcOH, AcOEt. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin.  $k = 2.6 \times 10^{-4}$  at 25°. *Et ester*: m.p. 194-5°.

**N-Chloroacetyl**: plates or prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 132-3°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>.  $k = 3.4 \times 10^{-4}$  at 25°. *Me ester*: prisms from ligroin. M.p. 59-60°.

**N-Bromoacetyl**: leaflets from H<sub>2</sub>O. M.p. 153° decomp.  $k = 3.4 \times 10^{-4}$  at 25°. *Me ester*: leaflets from ligroin. M.p. 71°.

**N-Benzoyl**: *N*-phenylhippuric acid. Amorph. M.p. 63°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

Wohl, Blank, D.R.P., 167,698, (*Chem. Zentr.*, 1906, I, 1069).

Mai, *Ber.*, 1902, 35, 580.

Curtius, *J. prakt. Chem.*, 1888, 38, 436.

Lippmann, D.R.P., 163,515, (*Chem. Zentr.*, 1905, II, 1475).

Bischoff, *Ber.*, 1889, 22, 1809.

Badische, D.R.P., 142,559, (*Chem. Zentr.*, 1903, II, 81).

Paal, Otten, *Ber.*, 1890, 23, 2592.

Fischer, Glud, *Ann.*, 1909, 369, 266.

Thorpe, Wood, *J. Chem. Soc.*, 1913, 103, 1606.

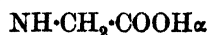
Stollé, *J. prakt. Chem.*, 1914, 90, 274.

Wessely, *Z. physiol. Chem.*, 1925, 146, 72.

B.D.C., E.P., 188,933, (*Chem. Abstracts*, 1923, 17, 1646).

Rebuffat, *Gazz. chim. ital.*, 1887, 17, 232.

**Phenylglycine-*o*-carboxylic Acid (Anthranilinoacetic acid)**



C<sub>8</sub>H<sub>6</sub>O<sub>4</sub>N

MW, 195

Needles from MeOH. M.p. 218-20°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Mod. sol. hot H<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Alc. sol. shows blue fluor. Intermediate in manufacture of indigo.

**$\alpha$ -Me ester**: C<sub>10</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 209. M.p. 160°.

**$\beta$ -Me ester**: m.p. 182°.  **$\alpha$ -Amide**: C<sub>10</sub>H<sub>12</sub>O<sub>3</sub>N<sub>2</sub>. MW, 208. M.p. 195°.  **$\alpha$ -Anilide**: m.p. 140-2°.

**Di-Me ester**: C<sub>11</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 223. Leaflets. M.p. 97°. Volatile in steam. C<sub>6</sub>H<sub>6</sub> and Et<sub>2</sub>O sols. show blue fluor. **N-Acetyl**: m.p. 83°. B.p. 205-12°/30 mm.

**$\alpha$ -Et ester**: C<sub>11</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 223. Needles from EtOH. M.p. 152°. Sol. CHCl<sub>3</sub>.  **$\beta$ -Me ester**: C<sub>12</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 237. M.p. 48°. **N-Acetyl**: m.p. 86-7°.  $k = 3.8 \times 10^{-4}$  at 25°. **N-Carboethoxyl**: m.p. 114-16°.

**$\beta$ -Et ester**: m.p. 182°.  **$\alpha$ -Amide**: C<sub>11</sub>H<sub>14</sub>O<sub>3</sub>N. MW, 222. Needles from EtOH. M.p. 180-2°.  **$\alpha$ -Nitrile**: C<sub>11</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 204. Cryst. from EtOH. M.p. 89°. Sol. C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.  **$\alpha$ -Anilide**: m.p. 164-6°. **N-Acetyl**: m.p. 130-2°. **N-Benzoyl**: m.p. 141-3°. **N-Carboethoxyl**: m.p. 106-8°.  **$\alpha$ -Hydrazide**: needles from EtOH. M.p. 166°.

**Di-Et ester**: C<sub>13</sub>H<sub>17</sub>O<sub>4</sub>N. MW, 251. M.p. 75°. **N-Acetyl**: plates or prisms. M.p. 63-4°. B.p. 214-18°/15 mm. decomp. **N-Benzoyl**: m.p. 53-4°. **N-Carboethoxyl**: m.p. 50°.

**$\alpha$ -Amide**: C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 194. M.p. 195°.

**Diamide**: C<sub>9</sub>H<sub>11</sub>O<sub>2</sub>N<sub>3</sub>. MW, 193. Leaflets from H<sub>2</sub>O. M.p. 198-200°.

**$\alpha$ -Nitrile**: C<sub>9</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub>. MW, 176. Leaflets from EtOH, needles from CHCl<sub>3</sub> or C<sub>6</sub>H<sub>6</sub>. M.p. 184°. Sol. Me<sub>2</sub>CO, Et<sub>2</sub>O. Mod. sol. boiling EtOH. Insol. C<sub>6</sub>H<sub>6</sub>. Sols. show violet fluor.  **$\beta$ -Me ester**: C<sub>10</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>. MW, 190. Needles from EtOH. M.p. 106-5°.

**$\beta$ -Nitrile**: cryst. M.p. 197° decomp.

**$\alpha$ -Anilide**: needles from EtOH. M.p. about 235° decomp.  **$\beta$ -Amide**: m.p. 185°.

**N-Me**: C<sub>10</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 209. Cryst. M.p. 189° decomp.

**N-Et**: C<sub>11</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 223. M.p. 184-6°.

**N-Phenyl**: C<sub>15</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 271. Prisms from MeOH.Aq. M.p. 165-7°.

**N-Benzyl**: C<sub>16</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 285. M.p. 190° decomp. **Di-Me ester**: C<sub>18</sub>H<sub>19</sub>O<sub>4</sub>N. MW, 313. M.p. 82-3°.

**N-Acetyl**: cryst. from MeOH or H<sub>2</sub>O. M.p. 214°.  $k = 1.05 \times 10^{-3}$  at 25°.

**N-Benzoyl**: cryst. from EtOH. M.p. 197°.

**$\alpha$ -Hydrazide**: needles from EtOH. M.p. 200-5°.

**Dihydrazide**: prisms from EtOH. M.p. 161°. Sol. EtOH. Spar. sol. H<sub>2</sub>O. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

pet. ether.  $B, 2HCl$ : m.p.  $201^{\circ}$ . *Dipicrate*: orange-red plates from  $H_2O$ . M.p.  $185^{\circ}$ .

v. Heyden, D.R.P., 138,207, (*Chem. Zentr.*, 1903, I, 304).

Leonhardt, D.R.P., 126,962, (*Chem. Zentr.*, 1902, I, 82).

Vorländer, *Ber.*, 1902, **35**, 1685.

Vorländer, Mumme, *Ber.*, 1902, **35**, 1699.

Kalle, D.R.P., 206,903, (*Chem. Zentr.*, 1909, I, 807).

Badische, D.R.P., 136,779, (*Chem. Zentr.*, 1902, II, 1351).

Vorländer, v. Schilling, *Ber.*, 1900, **33**, 554.

Houben, *Ber.*, 1913, **46**, 3998.

### Phenylglycine-*m*-carboxylic Acid.

*Diamide*: m.p.  $201-2^{\circ}$ . Sol. hot  $H_2O$ .

$\alpha$ -*Nitrile*: leaflets from 30% EtOH. M.p.  $193^{\circ}$ . Sol. hot  $H_2O$ , EtOH,  $Me_2CO$ ,  $C_6H_6$ .

Houben, Arnold, *Ber.*, 1908, **41**, 1573.

Lumière, Perrin, *Bull. soc. chim.*, 1903, **29**, 966.

### Phenylglycine-*p*-carboxylic Acid.

Cryst. from  $H_2O$ . M.p.  $219-21^{\circ}$  decomp. Spar. sol.  $H_2O$ .

$\alpha$ -*Amide*: prisms from EtOH. M.p.  $251^{\circ}$ . Spar. sol.  $H_2O$ . Insol.  $Et_2O$ ,  $C_6H_6$ .  $\beta$ -*Et ester*: needles from EtOH. M.p.  $142^{\circ}$ .

$\alpha$ -*Nitrile*: cryst. powder from  $Me_2CO$ . M.p.  $177^{\circ}$  decomp.

Einhorn, Seuffert, *Ber.*, 1910, **43**, 3001.

Mauthner, Suida, *Monatsh.*, 1890, **11**, 380.

Houben, Arnold, *Ber.*, 1908, **41**, 1572.

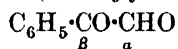
### Phenylglycollic Acid.

See Mandelic Acid.

### Phenylglycollytropine.

See Homatropine.

### Phenylglyoxal (*Benzoylformaldehyde*)



$C_8H_6O_2$  MW, 134

Needles +  $H_2O$  from  $H_2O$ . M.p.  $91^{\circ}$ . Sol. usual solvents. Sol. about 35 parts  $H_2O$  at  $20^{\circ}$ . Volatile in steam. Alkalis  $\rightarrow$  mandelic acid.  $KMnO_4 \rightarrow$  benzoic acid. Above m.p.  $\rightarrow$  anhydrous compound, a deep yellow oil, b.p.  $142^{\circ}/125$  mm.,  $120^{\circ}/50$  mm.

$\alpha$ -*Oxime*: see Isonitrosoacetophenone.

*Dioxime*: see Phenylglyoxime.

$\alpha$ -*Di-Me acetal*: b.p.  $247-8^{\circ}$ ,  $133-4^{\circ}/16$  mm.

$\alpha$ -*Di-Et acetal*: b.p.  $110^{\circ}/15$  mm.

$\alpha$ -*Hydrazone*: needles from  $C_6H_6$ . M.p.  $120-1^{\circ}$ . Mod. sol. EtOH, hot  $C_6H_6$ . Spar. sol.  $H_2O$ ,  $Et_2O$ .

$\alpha$ -*Semicarbazone*: pale yellowish leaflets from EtOH. M.p.  $217^{\circ}$  decomp.

$\alpha$ -*Thiosemicarbazone*: yellowish prisms from EtOH. M.p.  $170^{\circ}$ . Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ .

*Diphenylhydrazone*: phenylosazone, yellow leaflets or needles from EtOH. M.p.  $152^{\circ}$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

*Di-p-nitrophenylhydrazone*: m.p.  $310-11^{\circ}$  decomp.

Wieland, Semper, *Ann.*, 1908, **358**, 57.

Russanow, *Ber.*, 1891, **24**, 3501.

Wolff, Lindenhayn, *Ber.*, 1903, **36**, 4127.

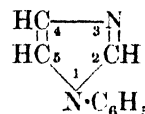
Wolff, *Ann.*, 1912, **394**, 34.

Henze, *Z. physiol. Chem.*, 1931, **198**, 84.

Madelung, Oberwegner, *Ber.*, 1932, **65**, 935.

Riley, Gray, *Organic Syntheses*, 1935, XV, 67.

### 1-Phenylglyoxaline (1-Phenyliminazole)



$C_9H_8N_2$

MW, 144

Cryst. M.p.  $13^{\circ}$ . B.p.  $276^{\circ}$ . Misc. with most ord. solvents. Insol.  $H_2O$ .

$B, 2HNO_3$ : prisms. M.p.  $82-6^{\circ}$ .

$B_2, H_2PtCl_6$ : reddish-yellow leaflets. M.p.  $203^{\circ}$  decomp. Mod. sol. hot  $H_2O$ .

*Picrate*: yellow needles from EtOH. M.p.  $152^{\circ}$ . Spar. sol.  $H_2O$ , cold EtOH.

Wohl, Marckwald, *Ber.*, 1889, **22**, 576, 1354.

Fischer, Hunsalz, *Ber.*, 1894, **27**, 2206.

### 2-Phenylglyoxaline (2-Phenyliminazole).

Leaflets from  $C_6H_6$ , needles from  $H_2O$ . M.p.  $148-9^{\circ}$ . B.p.  $340^{\circ}$ . Sol. EtOH. Spar. sol.  $H_2O$ ,  $C_6H_6$ .

$B, HNO_3$ : leaflets from EtOH. M.p.  $135^{\circ}$ .

*Acid oxalate*: needles from  $H_2O$ . M.p.  $219^{\circ}$ .

*Picrate*: needles from  $H_2O$ . M.p.  $238^{\circ}$ .

*N-Me*: see 1-Methyl-2-phenylglyoxaline.

Fargher, Pyman, *J. Chem. Soc.*, 1919, **115**, 232.

Maquenne, *Ann. chim. phys.*, 1891, **24**, 543.

### 4-Phenylglyoxaline (4-Phenyliminazole).

Leaflets from  $H_2O$ . M.p.  $128-9^{\circ}$ . Very sol. EtOH,  $Et_2O$ . Mod. sol. hot  $H_2O$ ,  $C_6H_6$ .

$B_2, H_2PtCl_6$ : orange-red prisms +  $3H_2O$ . M.p.  $215^{\circ}$  decomp.

Pinner, *Ber.*, 1902, **35**, 4135.

**Phenylglyoxime** (*Phenylglyoxal dioxime*) $\alpha$ -form $\text{C}_8\text{H}_8\text{O}_2\text{N}_2$  MW, 164

Needles from  $\text{CHCl}_3$  or  $\text{EtOH}$ . Aq. M.p.  $168^\circ$ .  
Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , acids. Spar. sol.  $\text{C}_6\text{H}_6$ . Insol. ligroin.

*N-Me*:  $\text{C}_9\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 178. M.p.  $209-10^\circ$ .  
Sol.  $\text{AcOH}$ , dil.  $\text{HCl}$ . Insol.  $\text{Na}_2\text{CO}_3$ . Gives greenish-yellow sol. in amyl alcohol or  $\text{NaOH}$ .

*O:N-Di-Me*:  $\text{C}_{10}\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 192. M.p.  $135^\circ$ . Sol. 20%  $\text{HCl}$ .

*Dipropionyl*: m.p.  $75^\circ$ .

*Diphenylurethane*: m.p.  $136^\circ$  decomp.

 $\beta$ -form

Needles from  $\text{EtOH}$ . M.p.  $180^\circ$ .  $\text{HCl}$  in  $\text{Et}_2\text{O}$  or  $\text{AcOH} \rightarrow \alpha$ -form.

*a-Me ether*:  $\text{C}_9\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 178. Prisms from ligroin. M.p.  $113^\circ$ . *b-Acetyl*: prisms from pet. ether. M.p.  $75^\circ$ .

*Di-Me ether*:  $\text{C}_{10}\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 192. Plates from  $\text{EtOH}$ . M.p.  $72^\circ$ . B.p.  $240^\circ/740$  mm.,  $137-8^\circ/40$  mm.

*Diacetyl*: m.p.  $71-2^\circ$ .

*Dipropionyl*: m.p.  $89-90^\circ$ .

*Dibenzoyl*: m.p.  $150^\circ$ .

*Diphenylurethane*: m.p.  $146-7^\circ$  decomp.

Longo, *Gazz. chim. ital.*, 1932, **62**, 139.

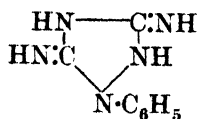
Ponzio, *Gazz. chim. ital.*, 1930, **60**, 825.

Avogadro, *Gazz. chim. ital.*, 1926, **56**, 713.

Ponzio, Avogadro, *Gazz. chim. ital.*, 1923, **53**, 25, 311.

**Phenylglyoxylic Acid.**

See Benzoylformic Acid.

**Phenylguanazole** $\text{C}_8\text{H}_9\text{N}_5$  MW, 175

Cryst. M.p.  $175^\circ$ . Sol.  $\text{EtOH}$ . Mod. sol.  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*B,HCl*: needles. M.p.  $240^\circ$ . Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*N-Nitroso deriv.*: yellow powder. M.p.  $245^\circ$ . Spar. sol.  $\text{EtOH}$ .

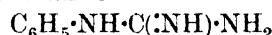
*N-Acetyl deriv.*: prisms from  $\text{H}_2\text{O}$ . M.p.  $244^\circ$ . Sol. warm  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ .

*Diacetyl deriv.*: prisms from  $\text{H}_2\text{O}$ . M.p.  $212^\circ$ .

*Tetra-acetyl*: needles. M.p.  $157^\circ$ .

Pellizzari, Roncagliolo, *Gazz. chim. ital.*, 1901, **31**, i, 477.

Pellizzari, *Gazz. chim. ital.*, 1891, **21**, ii, 146.

**Phenylguanidine** $\text{C}_7\text{H}_9\text{N}_3$  MW, 135

Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $66-7^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CCl}_4$ .

*B,HNO*: needles or prisms. M.p.  $128^\circ$  ( $118^\circ$ ).

*B,HBr*: m.p.  $71^\circ$ .

*B,H,PtCl*: m.p.  $197-8^\circ$ .

*B,H,SO*: m.p.  $205^\circ$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ .

*B,H,CO*: prisms from  $\text{H}_2\text{O}$ . M.p.  $138-40^\circ$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

*Benzoyl deriv.*: pale yellow prisms from  $\text{C}_6\text{H}_6$ -pet. ether. M.p.  $91-2^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ .

*Picrate*: yellow prisms from  $\text{EtOH}$ . M.p.  $186^\circ$ .

*Dibenzoyl deriv.*: needles from  $\text{EtOH}$ . M.p.  $187^\circ$ .

*Picrate*: needles. M.p.  $223^\circ$  ( $218-20^\circ$ ).

Kampf, *Ber.*, 1904, **37**, 1683.

Wheeler, Johnson, *Am. Chem. J.*, 1901, **26**, 417.

Smith, *J. Am. Chem. Soc.*, 1929, **51**, 477.

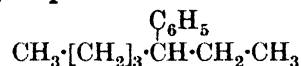
Schering-Kahlbaum A.-G., D.R.P., 565,881, (*Chem. Zentr.*, 1933, I, 1685).

**N-Phenylheptadecylamine.**

See Heptadecylaniline.

**1-Phenylheptane.**

See *n*-Heptylbenzene.

**3-Phenylheptane** $\text{C}_{13}\text{H}_{20}$  MW, 176*d.*

B.p.  $112^\circ/15$  mm.  $D_4^{25}$  0.856.  $[\alpha]_D^{25} + 0.97^\circ$ .

*dl.*

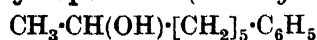
Obtained by cracking gasoline. B.p.  $68-71^\circ/3$  mm.  $n_D^{20}$  1.4871.

Levene, Marker, *J. Biol. Chem.*, 1932, **97**, 563.

Tilicheev, Kuruindin, *Chem. Abstracts*, 1931, **25**, 3469.

**Phenylheptanol-1.**

See Phenylheptyl Alcohol.

**1-Phenylheptanol-6** (*7-Phenylheptanol-2*) $\text{C}_{13}\text{H}_{20}\text{O}$  MW, 192

B.p. 169°/50 mm. (164°/23 mm.).  $D_4^{25}$  0.9482 (0.9389).  $n_D^{25}$  1.50575 (1.5028).

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 155.

Davies, Dixon, Jones, *J. Chem. Soc.*, 1930, 471.

## 2-Phenylheptanol-3

$\text{CH}_3 \cdot [\text{CH}_2]_3 \cdot \text{CH}(\text{OH}) \cdot \text{CH} \cdot \text{C}_6\text{H}_5$   
 $\text{C}_{13}\text{H}_{20}\text{O}$  MW, 192

B.p. 156–60°/30 mm.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

## 3-Phenylheptanol-4

$\text{CH}_3 \cdot [\text{CH}_2]_2 \cdot \text{CH}(\text{OH}) \cdot \text{CH} \cdot \text{C}_6\text{H}_5$   
 $\text{C}_{13}\text{H}_{20}\text{O}$  MW, 192

B.p. 145–7°/20 mm.

See previous reference.

**4-Phenylheptanol-4** (*Diethylphenylcarbinol*, *ω*-*diethylbenzyl alcohol*)

$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C}(\text{OH}) \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH}_3$   
 $\text{C}_{13}\text{H}_{20}\text{O}$  MW, 192

B.p. 134°/26 mm.  $D_4^0$  0.9589,  $D_4^{15}$  0.9470.  $n_D^{15}$  1.516.

*Acetyl*: b.p. 160°/19 mm.  $D_4^{15}$  0.8973.

Amouroux, Murat, *Compt. rend.*, 1912, **154**, 993.

Gilman, Fothergill, Parker, *Rec. trav. chim.*, 1929, **48**, 748.

## 2-Phenylheptanone-3

$\text{CH}_3 \cdot [\text{CH}_2]_3 \cdot \text{CO} \cdot \text{CH} \cdot \text{C}_6\text{H}_5$   
 $\text{C}_{13}\text{H}_{18}\text{O}$  MW, 190

B.p. 256–7°.

*Semicarbazone*: m.p. 129°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

## 2-Phenylheptanone-6

$\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{C}_6\text{H}_5$   
 $\text{H}_{18}\text{O}$  MW, 190

B.p. 136–40°/13 mm.  $D_4^{20}$  0.9629.

*p*-*Nitrophenylhydrazone*: m.p. 117°.

Nenitzescu, Gavát, *Ann.*, 1935, **519**, 270.

## 3-Phenylheptanone-2

$\text{CH}_3 \cdot [\text{CH}_2]_3 \cdot \text{CH} \cdot \text{CO} \cdot \text{CH}_3$   
 $\text{C}_{13}\text{H}_{18}\text{O}$  MW, 190

B.p. 250–1°.  $D_4^0$  0.960.

*Oxime*: m.p. 63–6°.

*Semicarbazone*: m.p. 156–8°.

Tiffeneau, Lévy, Jullien, *Bull. soc. chim.*, 1931, **49**, 1788.

## 3-Phenylheptanone-4

$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CO} \cdot \text{CH} \cdot \text{C}_6\text{H}_5$   
 $\text{C}_{13}\text{H}_{18}\text{O}$  MW, 190

B.p. 242–5°.

*Semicarbazone*: m.p. 106–7°.

See previous reference.

## 4-Phenylheptanone-3

$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{CH}_3$   
 $\text{C}_{13}\text{H}_{18}\text{O}$  MW, 190

B.p. 240–5°.

*Semicarbazone*: m.p. 108–9°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

**4-Phenyl-3-heptene** (*1-Propyl-1-phenyl-butylene-1*, *4-phenyl-3-heptylene*)

$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} \cdot \text{CH} \cdot \text{CH}_2 \cdot \text{CH}_3$   
 $\text{C}_{13}\text{H}_{18}$  MW, 174

B.p. 228°/760 mm.  $D_4^{25}$  0.8855.  $n_D^{15}$  1.522.

Amouroux, Murat, *Compt. rend.*, 1912, **154**, 993.

**1-Phenylheptyl Alcohol** (*n-Hexylphenylcarbinol*, *1-phenylheptanol-1*)

$\text{CH}_3 \cdot [\text{CH}_2]_5 \cdot \text{CH}(\text{OH}) \cdot \text{C}_6\text{H}_5$   
 $\text{C}_{13}\text{H}_{20}\text{O}$  MW, 192

B.p. 275°, 176°/40 mm., 156°/25 mm.

*Phenylurethane*: m.p. 77°.

Colacicchi, *Atti accad. Lincei*, 1910, **19**, ii, 601.

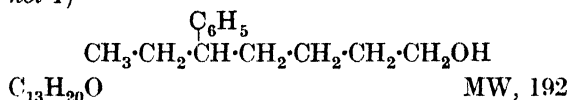
**3-Phenylheptyl Alcohol** (*3-Phenylheptanol-1*)

$\text{CH}_3 \cdot [\text{CH}_2]_3 \cdot \text{CH} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{OH}$   
 $\text{C}_{13}\text{H}_{20}\text{O}$  MW, 192

*l.*

B.p. 150°/10 mm.  $D_4^{27}$  0.947.  $[\alpha]_D^{27}$  –1.45°.

dl.

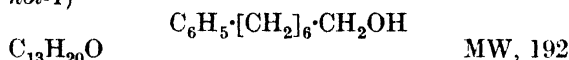
B.p. 116–20°/3 mm.  $D_{25}^{25}$  0.9466.  $n_D^{20}$  1.5070.Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, **54**, 2525.Levene, Marker, *J. Biol. Chem.*, 1932, **97**, 563.**5-Phenylheptyl Alcohol** (5-Phenylheptanol-1)

d.

B.p. 145°/1 mm.  $D_4^{25}$  0.952.  $[\alpha]_D^{25} + 1.47^\circ$ .

l.

B.p. 123°/1 mm.

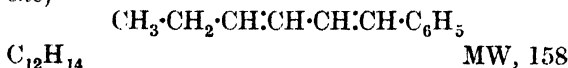
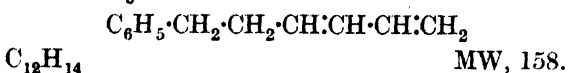
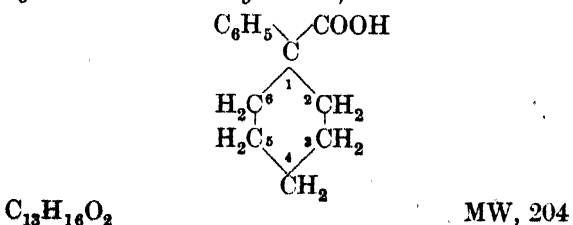
Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 749; 1935, **110**, 329.**7-Phenylheptyl Alcohol** (7-Phenylheptanol-1)

Liq. with faint odour of roses. B.p. 170–2°/15 mm.

Acetyl: b.p. 188–90°/24 mm.

v. Braun, *Ber.*, 1911, **44**, 2878.**Phenylheptylene.**

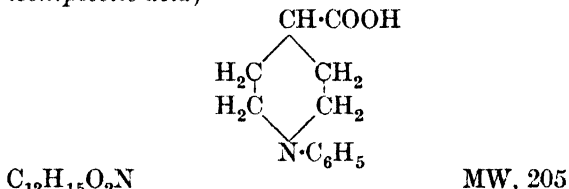
See Phenylheptene.

**1-Phenyl-1 : 3-hexadiene** (1-Styryl-1-butylene)Oil B.p. 128°/16 mm.  $D_4^{15}$  0.9253.  $n_D^{15}$  1.60252. Na + EtOH  $\longrightarrow$  1-phenyl-2-hexene.Klages, *Ber.*, 1907, **40**, 1770.**6-Phenyl-1 : 3-hexadiene**B.p. 99.5–100.5°/11 mm.  $D_4^{15}$  0.9304.  $n_D^{15}$  1.5446.Cohen, *J. Chem. Soc.*, 1935, 433.**1-Phenylhexahydrobenzoic Acid** (1-Phenylcyclohexane-1-carboxylic acid)

Cryst. M.p. 121°.

Amide:  $\text{C}_{13}\text{H}_{17}\text{ON}$ . MW, 203. Cryst. from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 95–6°.Case, *J. Am. Chem. Soc.*, 1934, **56**, 716.**2-Phenylhexahydrobenzoic Acid** (2-Phenylcyclohexane-1-carboxylic acid).Prisms from pet. ether. M.p. 105–7° (104–5°). Sol. MeOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ , warm pet. ether. Spar. sol.  $\text{H}_2\text{O}$ .Kipping, Perkin, *J. Chem. Soc.*, 1890, **57**, 316.Ranado, Léon, *Chem. Abstracts*, 1927, **21**, 1108.Cook, Hewett, Lawrence, *J. Chem. Soc.*, 1936, 70.**4-Phenylhexahydrobenzoic Acid** (4-Phenylcyclohexane-1-carboxylic acid).

Exists in two forms.

(i) Leaflets from 50% AcOH. M.p. 204° (202°). Fuming HCl at 170°  $\longrightarrow$  (ii).Me ester:  $\text{C}_{14}\text{H}_{18}\text{O}_2$ . MW, 218. M.p. 28°. B.p. 172–3°/15 mm.(ii) Needles from 40% EtOH. M.p. 113°. Sol. EtOH,  $\text{CHCl}_3$ , AcOH. Less sol.  $\text{Et}_2\text{O}$ , pet. ether. Sol. 1000 parts boiling  $\text{H}_2\text{O}$ . Conc. HCl at 180°  $\longrightarrow$  (i).Rassow, *Ann.*, 1894, **282**, 147.Nenitzescu, Gavát, *Ann.*, 1935, **519**, 266.**N-Phenylhexahydroisonicotinic Acid** (N-Phenylpiperidine-4-carboxylic acid, N-phenylisonipicotic acid)

Cryst. from EtOH. M.p. 131°.

B, HBr: cryst. from  $\text{H}_2\text{O}$ . M.p. 218–19°.Me ester:  $\text{C}_{13}\text{H}_{17}\text{O}_2\text{N}$ . MW, 219. Cryst. from ligroin. M.p. 46°.

Anilide: m.p. 210°.

Methiodide: decomp. 180°.

Picrate: cryst. from MeOH. M.p. 207° decomp.

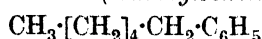
Prelog, Hanousek, *Chem. Abstracts*, 1934, **28**, 5825.**Phenylhexahydrophenol.**

See Phenylcyclohexanol.

**Phenyl hexahydrostyryl Ketone.**

See Hexahydrobenzylideneacetophenone.

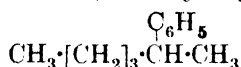


**1-Phenylhexane** (*n-Hexylbenzene*)

$\text{C}_{12}\text{H}_{18}$  MW, 162

B.p. 219–20° (215°).  $D_4^{20}$  0.8613.  $n_D^{20}$  1.490.

v. Braun, Deutsch, *Ber.*, 1912, **45**, 2180.  
Sabatier, Mailhe, *Compt. rend.*, 1914, **158**, 834.

**2-Phenylhexane** ( $\omega$ -Methylbutyltoluene)

$\text{C}_{12}\text{H}_{18}$  MW, 162

*d.*

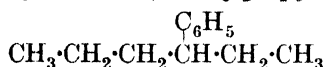
B.p. 100°/22 mm.  $D_4^{25}$  0.855.  $[\alpha]_D^{25} + 1.96^\circ$ .

*dl.*

B.p. 208°/760 mm.  $D_4^{15}$  0.869.  $n^{15}$  1.492.

Brochet, *Bull. soc. chim.*, 1893, **9**, 687.

Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 749.

**3-Phenylhexane** ( $\omega$ -Ethylpropyltoluene)

$\text{C}_{12}\text{H}_{18}$  MW, 162

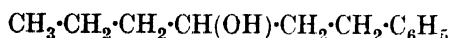
*d.*

B.p. 103°/25 mm.  $D_4^{24}$  0.863.  $[\alpha]_D^{24} + 0.57^\circ$ .

See last reference above.

**Phenylhexanol-1.**

See Phenylhexyl Alcohol.

**1-Phenylhexanol-3** (*Propyl- $\beta$ -phenylethyl-carbinol*)

$\text{C}_{12}\text{H}_{18}\text{O}$  MW, 178

*d.*

Needles. M.p. 34°. B.p. 146°/16 mm.  
Volatile in steam.

*Acid phthalate*: m.p. 75°.  $[\alpha]_{5893} + 25.5^\circ$ .  
*Brucine salt*: cryst. from  $\text{Me}_2\text{CO}$ . M.p. 75°.  
 $[\alpha]_{5893} - 12.5^\circ$ .

*l.*

Needles. M.p. 34°. B.p. 146°/16 mm.  
Volatile in steam.

*Acid phthalate*: m.p. 75°.  $[\alpha]_{5893} - 25.5^\circ$ .  
*Brucine salt*: cryst. from  $\text{Me}_2\text{CO}$ . M.p. 95°.  
 $[\alpha]_{5893} - 2.1^\circ$  in EtOH.

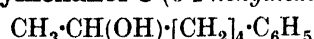
*Formyl*: b.p. 147°/18 mm.  $D_4^{20}$  0.9872.

*Acetyl*: b.p. 154°/20 mm.  $D_4^{20}$  0.9725.

*dl.*

*Acid phthalate*: prisms from EtOH. M.p. 108°.

Hewitt, Kenyon, *J. Chem. Soc.*, 1925, 1094.

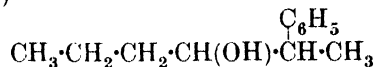
**1-Phenylhexanol-5** (*6-Phenylhexanol-2*)

$\text{C}_{12}\text{H}_{18}\text{O}$  MW, 178

B.p. 148°/18 mm.  $D_4^{20}$  0.9567.  $n_D^{20}$  1.50787.

*Phenylurethane*: needles from pet. ether.  
M.p. 65°.

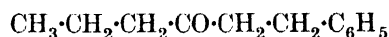
Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 151.

**2-Phenylhexanol-3** (*Propyl- $\alpha$ -phenylethyl-carbinol*)

$\text{C}_{12}\text{H}_{18}\text{O}$  MW, 178

B.p. 150–5°/30 mm.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

**1-Phenylhexanone-3** (*Propyl  $\beta$ -phenylethyl ketone*)

$\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176

B.p. 133–5°/8 mm.

*Oxime*: m.p. 43°.

*Semicarbazone*: cryst. from  $\text{C}_6\text{H}_6$ . M.p. 79°.

Rupe, Hirschmann, *Helv. Chim. Acta*, 1931, **14**, 698.

**1-Phenylhexanone-4** (*6-Phenylhexanone-3*)

$\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176

Colourless oil. B.p. 137–40°/10 mm.

*Semicarbazone*: cryst. from EtOH. M.p. 149°.

Rupe, Hirschmann, *Helv. Chim. Acta*, 1931, **14**, 700.

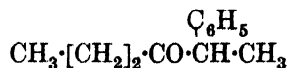
**1-Phenylhexanone-5** (*6-Phenylhexanone-2*)

$\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176

B.p. 137°/8 mm.

*Semicarbazone*: m.p. 143°.

Ramart-Lucas, Labaune, *Ann. chim.*, 1931, **16**, 276.

**2-Phenylhexanone-3** (*Propyl  $\alpha$ -phenylethyl ketone*)

$\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176

B.p. 240–2°.

*Semicarbazone*: m.p. 148–9°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

**2-Phenylhexanone-5** (5-Phenylhexanone-2)

$$\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \overset{\text{C}_6\text{H}_5}{\text{CH}} \cdot \text{CH}_3$$
 $\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176  
 B.p. 122–5°/9 mm.  
*Semicarbazone*: m.p. 147°.  
 Nenitzescu, Gavát, *Ann.*, 1935, **519**, 270.

**3-Phenylhexanone-2**

$$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \overset{\text{C}_6\text{H}_5}{\text{CH}} \cdot \text{CO} \cdot \text{CH}_3$$
 $\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176  
 B.p. 235–6°.  $D_4^{20}$  0.970.  
*Oxime*: m.p. 42–3°.  
*Semicarbazone*: m.p. 130–1°.  
 Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

**3-Phenylhexanone-4** (4-Phenylhexanone-3)

$$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CO} \cdot \overset{\text{C}_6\text{H}_5}{\text{CH}} \cdot \text{CH}_2 \cdot \text{CH}_3$$
 $\text{C}_{12}\text{H}_{16}\text{O}$  MW, 176  
 Yellow oil. B.p. 114–16°/13 mm.  $D_4^{20}$  0.965,  $D_4^{25}$  0.978.  
*Oxime*: m.p. 57–8°.  
*Semicarbazone*: m.p. 139–40°. Mod. sol. EtOH,  $\text{C}_6\text{H}_6$ .  
 Tiffeneau, Lévy, *Bull. soc. chim.*, 1923, **33**, 735.

**6-Phenylhexene-1**

$$\text{C}_6\text{H}_5 \cdot [\text{CH}_2]_3 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{CH}_2$$
 $\text{C}_{12}\text{H}_{16}$  MW, 160  
 B.p. 94–5°/10 mm.  $D_4^{20}$  0.8839.  $n_D^{20}$  1.5033.  
 v. Braun, Deutsch, Schmatlock, *Ber.*, 1912, **45**, 1257.

**1-Phenylhexene-2** (sym.-Propylbenzylethyl-ene, 1-propyl-3-phenylpropylene)

$$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{CH} \cdot \text{CH}_2 \cdot \text{C}_6\text{H}_5$$
 $\text{C}_{12}\text{H}_{16}$  MW, 160  
 B.p. 108°/16 mm.  $D_4^{16}$  0.8898.  $n_D^{16}$  1.5058.  
 Klages, *Ber.*, 1907, **40**, 1771.

**2-Phenylhexene-3**

$$\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{CH} \cdot \overset{\text{C}_6\text{H}_5}{\text{CH}} \cdot \text{CH}_3$$
 $\text{C}_{12}\text{H}_{16}$  MW, 160  
 B.p. 84°/10 mm.  
 Riiber, *Ber.*, 1903, **36**, 1405.  
 Dict. of Org. Comp.—III.

**1-Phenyl-1-hexenone-5**

$$\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{CH} \cdot \text{C}_6\text{H}_5$$
 $\text{C}_{12}\text{H}_{14}\text{O}$  MW, 174  
 B.p. 153–5°/10 mm.  
*Semicarbazone*: cryst. from AcOEt. M.p. 132°.  
 Fischer, Wiedemann, *Ann.*, 1935, **520**, 69.

**3-Phenyl-2-hexenone-5**

$$\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_2 \cdot \overset{\text{C}_6\text{H}_5}{\text{C}} \cdot \text{CH} \cdot \text{CH}_3$$
 $\text{C}_{12}\text{H}_{14}\text{O}$  MW, 174  
 B.p. 138°/14 mm.  
*Semicarbazone*: plates from EtOH. M.p. 185°.  
 Johnson, Kon, *J. Chem. Soc.*, 1926, 2758.

**3-Phenyl-3-hexenone-5**

$$\text{CH}_3 \cdot \text{CO} \cdot \text{CH} \cdot \overset{\text{C}_6\text{H}_5}{\text{C}} \cdot \text{CH}_2 \cdot \text{CH}_3$$
 $\text{C}_{12}\text{H}_{14}\text{O}$  MW, 174  
 B.p. 138°/14 mm.  
*Semicarbazone*: plates. M.p. 158°.  
 See previous reference.

**1-Phenylhexyl Alcohol** (1-Phenylhexanol-1, n-amylphenylcarbinol)

$$\text{CH}_3 \cdot [\text{CH}_2]_4 \cdot \text{CH}(\text{OH}) \cdot \text{C}_6\text{H}_5$$
 $\text{C}_{12}\text{H}_{18}\text{O}$  MW, 178  
 Viscous liq. B.p. 170°/50 mm.  $D_4^{25}$  0.9477.  $n_D^{25}$  1.5042.  
 Davies, Dixon, Jones, *J. Chem. Soc.*, 1930, 470.

**2-Phenylhexyl Alcohol** (2-Phenylhexanol-1)

$$\text{CH}_3 \cdot [\text{CH}_2]_3 \cdot \overset{\text{C}_6\text{H}_5}{\text{CH}} \cdot \text{CH}_2 \cdot \text{OH}$$
 $\text{C}_{12}\text{H}_{18}\text{O}$  MW, 178  
*d.*  
 B.p. 127°/1 mm.  $D_4^{25}$  0.967.  $[\alpha]_D^{25} + 1.96^\circ$ .  
 Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 772.

**3-Phenylhexyl Alcohol** (3-Phenylhexanol-1)

$$\text{CH}_3 \cdot [\text{CH}_2]_2 \cdot \overset{\text{C}_6\text{H}_5}{\text{CH}} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{OH}$$
 $\text{C}_{12}\text{H}_{18}\text{O}$  MW, 178  
*d.*  
 B.p. 127°/5 mm.  $D_4^{25}$  0.955.  $n_D^{25}$  1.5101.  $[\alpha]_D^{25} + 2.51^\circ$ .  
 Levene, Marker, *J. Biol. Chem.*, 1931, **93**, 765.

**4-Phenylhexyl Alcohol (4-Phenylhexanol-1)**

$$\text{C}_6\text{H}_5$$

$$\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}\cdot[\text{CH}_2]_2\cdot\text{CH}_2\text{OH}$$

$$\text{C}_{12}\text{H}_{18}\text{O}$$
 MW, 178  
*d.*  
 B.p. 125°/5 mm.  $[\alpha]_D^{25} + 0.21^\circ$ .  
 Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 335.

**6-Phenylhexyl Alcohol (6-Phenylhexanol-1)**

$$\text{C}_6\text{H}_5\cdot[\text{CH}_2]_5\cdot\text{CH}_2\text{OH}$$

$$\text{C}_{12}\text{H}_{18}\text{O}$$
 MW, 178  
 B.p. 160–1°/13 mm.  
*Me ether*:  $\text{C}_{13}\text{H}_{20}\text{O}$ . MW, 192. B.p. 140°/13 mm.  
*Acetyl*: b.p. 166–8°/13 mm.  
 v. Braun, *Ber.*, 1911, **44**, 2876.  
 v. Braun, Deutsch, *Ber.*, 1912, **45**, 2177.

**1-Phenylhippuric Acid (1-Benzoylamino-phenylacetic acid)**

$$\text{C}_6\text{H}_5$$

$$\text{C}_6\text{H}_5\cdot\text{CO}\cdot\text{NH}\cdot\text{CH}\cdot\text{COOH}$$

$$\text{C}_{15}\text{H}_{13}\text{O}_3\text{N}$$
 MW, 255  
 Needles from EtOH. M.p. 175.5°. Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O.  
*Et ester*:  $\text{C}_{17}\text{H}_{17}\text{O}_3\text{N}$ . MW, 283. Cryst. M.p. 84°.  
*Phenyl ester*:  $\text{C}_{21}\text{H}_{17}\text{O}_3\text{N}$ . MW, 331. Needles from EtOH. M.p. 131°.  
 Bayer, D.R.P., 55,026.  
 Kossel, *Ber.*, 1891, **24**, 4151.

**N-Phenylhippuric Acid.**

*See under Phenylglycine.*

**1-Phenylhydantoic Acid (α-Ureido-phenylacetic acid)**

$$\text{C}_6\text{H}_5$$

$$\text{H}_2\text{N}\cdot\text{CO}\cdot\text{NH}\cdot\text{CH}\cdot\text{COOH}$$

$$\text{C}_9\text{H}_{10}\text{O}_3\text{N}_2$$
 MW, 194  
 Prisms from H<sub>2</sub>O. M.p. 196–196.5° decomp. (178°). Spar. sol. cold H<sub>2</sub>O.  
*Et ester*:  $\text{C}_{11}\text{H}_{14}\text{O}_3\text{N}_2$ . MW, 222. Cryst. M.p. 139–40°. Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O.  
*Amide*:  $\text{C}_9\text{H}_{11}\text{O}_3\text{N}_3$ . MW, 193. Prisms from EtOH.Aq. M.p. 223° decomp. Sol. EtOH, hot H<sub>2</sub>O. Less sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Pinner, *Ber.*, 1888, **21**, 2326.

Dakin, Dudley, *J. Biol. Chem.*, 1914, **18**, 49.

Pinner, Spilker, *Ber.*, 1889, **22**, 697.

Kossel, *Ber.*, 1891, **24**, 4150.

**4-Phenylhydantoic Acid**

$$\text{C}_6\text{H}_5\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}\cdot\text{CH}_2\cdot\text{COOH}$$

$$\text{C}_9\text{H}_{10}\text{O}_3\text{N}_2$$
 MW, 194  
 Needles. M.p. 197° decomp.  
*Et ester*: needles. M.p. 108–9°.

Bailey, *Am. Chem. J.*, 1902, **28**, 386.

**1-Phenylhydantoin**

$$\text{C}_6\text{H}_5\cdot\text{N}_1\text{---CH}_2$$

$$\text{OC}^2$$

$$\text{HN}^3\text{---CO}^4$$

$$\text{C}_9\text{H}_8\text{O}_2\text{N}_2$$
 MW, 176  
 Fine needles. M.p. 193–4°. Mod. sol. EtOH.  
 Spar. sol. H<sub>2</sub>O.

Schwebel, *Ber.*, 1877, **10**, 2049.

Friedrich, Beckurts, *Arch. Pharm.*, 1899, **237**, 337.

**3-Phenylhydantoin.**

Needles from H<sub>2</sub>O. M.p. 159–60°. Sol. EtOH, Me<sub>2</sub>CO, hot C<sub>6</sub>H<sub>6</sub>, conc. min. acids.  
 Spar. sol. Et<sub>2</sub>O.

Mouneyrat, *Ber.*, 1900, **33**, 2394.

**5-Phenylhydantoin.**

Needles. M.p. 178°. Sol. EtOH, alkalis.  
 Spar. sol. H<sub>2</sub>O.  
*N-Me*:  $\text{C}_{10}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 190. Needles from H<sub>2</sub>O. M.p. 161°. Spar. sol. cold EtOH.  
*N-Et*: prisms from H<sub>2</sub>O. M.p. 94°. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.  
*N-Acetyl*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 145°.

Pinner, Lifschütz, *Ber.*, 1887, **20**, 2355.

Pinner, *Ber.*, 1888, **21**, 2325.

Lehmann, *Ber.*, 1901, **34**, 372.

Bergs, D.R.P., 566,094, (*Chem. Abstracts*, 1933, **27**, 1001).

**1-Phenylhydracrylic Acid.**

*See Tropic Acid.*

**2-Phenylhydracrylic Acid.**

*See β-Hydroxyhydrocinnamic Acid.*

**α-Phenylhydratropic Acid.**

*See 1:1-Diphenylpropionic Acid.*

**Phenylhydrazidine (Acetylamidrazone)**

$$\text{CH}_3\cdot\text{CO}\cdot\underset{\gamma}{\text{C}}(\text{NH}_2)_2\cdot\underset{\beta}{\text{N}}\cdot\underset{\alpha}{\text{NH}}\cdot\text{C}_6\text{H}_5$$

$$\text{C}_9\text{H}_{11}\text{ON}_3$$
 MW, 177  
 Yellow needles. M.p. 183°. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin, boiling H<sub>2</sub>O.

$\gamma$ -N-Acetyl: needles. M.p. 143°. Very sol.  $\text{CHCl}_3$ . Mod. sol.  $\text{Et}_2\text{O}$ . Insol. ligroin.

Phenylhydrazone: cryst. powder or needles. M.p. 224°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ .  $B, \text{HNO}_2$ : needles. M.p. 135°.

Bamberger, Lorenzen, *Ber.*, 1892, **25**, 3541.

Bamberger, Gruyter, *Ber.*, 1893, **26**, 2785; *J. prakt. Chem.*, 1901, **64**, 234.

### Phenylhydrazine (Hydrazinobenzene)



MW, 108

Plates or prisms. M.p. 23°. B.p. 241–2°, 137–8°/18 mm.  $D_4^{20}$ : 1.0978.  $n_D^{20}$ : 1.60813. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin. Heat of comb.  $C_v$  879.2 Cal.  $k = 1.62 \times 10^{-9}$  at 15°. Weak base.

$B, \text{HF}$ : leaflets. M.p. 166–7° decomp.

$B, \text{HCl}$ : leaflets from  $\text{EtOH}$ . M.p. 240°.

$B, \text{HNO}_3$ : leaflets. M.p. 145°.

$N'$ -Phenyl: see Hydrazobenzene.

$N'$ -Formyl: leaflets from  $\text{EtOH}$ . M.p. 145°. Sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

$N$ -Acetyl: leaflets from  $\text{C}_6\text{H}_6$ -ligroin. M.p. 125–6°. Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ , ligroin.  $N'$ -Formyl: cryst. M.p. 77–8°.

$N$ -Acetyl: prisms. M.p. 129°. Sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Reduces Fehling's.  $N$ -Formyl: cryst. M.p. 86°.

$N$ :  $N'$ -Diacetyl: plates or needles from  $\text{EtOH}$ - $\text{C}_6\text{H}_6$ . M.p. 107–8°. Very sol. boiling  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ . Reduces cold Fehling's.

$N'$ -Propionyl: plates from  $\text{CHCl}_3$ . M.p. 157–8°.

$N'$ -Butyryl: plates from  $\text{H}_2\text{O}$ . M.p. 103–4°.

$N$ -Isobutyryl: plates or needles from pet. ether. M.p. 46–8°.

$N'$ -Isobutyryl: leaflets from  $\text{H}_2\text{O}$ . M.p. 142–3°.

$N'$ -Isovaleryl: leaflets from 30%  $\text{EtOH}$ . M.p. 112–112.5°.

$N$ -Benzoyl: needles from  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . Aq. M.p. 70°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{H}_2\text{O}$ . Reduces warm Fehling's.

$N'$ -Benzoyl: prisms from  $\text{EtOH}$ . M.p. 168°. Mod. sol. hot  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ .

$N$ :  $N'$ -Dibenzoyl: prisms from  $\text{EtOH}$ . M.p. 177–8°. Mod. sol. hot  $\text{EtOH}$ . Spar. sol.  $\text{H}_2\text{O}$ . Reduces  $\text{NH}_3$ ,  $\text{AgNO}_3$ .

$N$ -Nitroso:  $\text{C}_6\text{H}_5\text{N}(\text{NO})\cdot\text{NH}_2$ .  $\text{C}_6\text{H}_7\text{ON}_3$ . MW,

137. Yellow cryst. M.p. 51°. Dil.  $\text{HCl} \rightarrow$  phenyl azide. Poisonous. Forms red  $\text{Cu}$  salt.

Michaelis, Schmidt, *Ann.*, 1889, **252**, 302.

Leighton, *Am. Chem. J.*, 1898, **20**, 677.

Widman, *Ber.*, 1894, **27**, 2964.

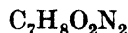
Thompson, *J. Soc. Dyers Colourists*, 1921, **37**, 7.

Coleman, *Organic Syntheses*, Collective Vol. I, 432.

Wurster, *Ber.*, 1887, **20**, 2633.

Bamberger, Hauser, *Ann.*, 1910, **375**, 317.

### Phenylhydrazine- $\alpha$ -carboxylic Acid ( $\alpha$ -Phenylhydraziniformic acid)



MW, 152

*Me ester*:  $\text{C}_8\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 166. Rhombic prisms from pet. ether. M.p. 69–70°.

*Et ester*:  $\text{C}_9\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 180. Plates. M.p. 24–5°. B.p. 157°/15 mm. Spar. sol. usual solvents. Alc.  $\text{FeCl}_3 \rightarrow$  red col.  $B, \text{HCl}$ : needles. M.p. 157–8°.

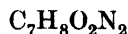
*Amide*: see 2-Phenylsemicarbazide.

Rupe, Gebhardt, *Ber.*, 1899, **32**, 11.

Busch, Limpach, *Ber.*, 1911, **44**, 1583.

Willstätter, Ulbrich, Pogány, Maimeri, *Ann.*, 1929, **477**, 168.

### Phenylhydrazine- $\beta$ -carboxylic Acid ( $\omega$ -Phenylcarbazinic acid, $\beta$ -phenylhydraziniformic acid)



MW, 152

Free acid not isolated.

*K salt*: needles from  $\text{EtOH}$ . M.p. 243° decomp. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*Me ester*:  $\text{C}_8\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 166. Prisms from  $\text{H}_2\text{O}$ . M.p. 115–17°.

*Et ester*:  $\text{C}_9\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 180. Cryst. from  $\text{EtOH}$ . Aq. M.p. 82–3°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , hot  $\text{H}_2\text{O}$ . Conc.  $\text{H}_2\text{SO}_4$  sol. with  $\text{FeCl}_3 \rightarrow$  intense red col.

*Phenyl ester*:  $\text{C}_{13}\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 228. Needles. M.p. 122–3°.

*Amide*: see 1-Phenylsemicarbazide.

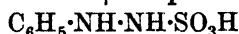
$B, \text{C}_6\text{H}_5\cdot\text{NH}\cdot\text{NH}_2$ : cryst. powder. M.p. 80° decomp.

Busch, Limpach, *Ber.*, 1911, **44**, 1582.

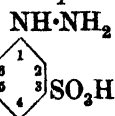
Freundler, *Bull. soc. chim.*, 1901, **25**, 859.

Busch, Stern, *J. prakt. Chem.*, 1899, **60**, 236.

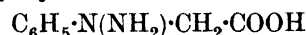
Heller, *Ann.*, 1891, **263**, 281.

**Phenylhydrazine-*o*-carboxylic Acid**  
(*o*-Hydrazinobenzoic acid) $C_7H_8O_2N_2$  MW, 152Needles from  $H_2O$ . M.p.  $249^\circ$ . Mod. sol. hot  $H_2O$ . Spar. sol. EtOH,  $Et_2O$ . Reduces Fehling's and  $NH_3 \cdot AgNO_3$  in the cold.*B, HBr*: needles. M.p.  $207-10^\circ$ .*Nitrile*: *o*-cyanophenylhydrazine.  $C_7H_7N_3$ . MW, 133. Leaflets from  $H_2O$ . M.p.  $152-3^\circ$ . Sol. EtOH,  $Et_2O$ . Insol. ligroin. *B, HCl*: needles. M.p.  $160-1^\circ$ .  $B_2H_2SO_4$ : m.p.  $215-16^\circ$ . *Picrate*: needles. M.p.  $238^\circ$ . $\alpha$ -*N-Me*:  $C_8H_{10}O_2N_2$ . MW, 166. Cryst. from ligroin. M.p.  $120^\circ$ . Very sol. EtOH.Fischer, *Ber.*, 1880, 13, 680.Acree, *Am. Chem. J.*, 1907, 37, 365.Gabriel, *Ber.*, 1903, 36, 805.**Phenylhydrazine-*m*-carboxylic Acid**  
(*m*-Hydrazinobenzoic acid).Pale yellow leaflets. M.p.  $186^\circ$  decomp. Spar. sol. EtOH, hot  $H_2O$ . Insol.  $Et_2O$ . Reacts strongly acid. Reduces Fehling's.Roder, *Ann.*, 1886, 236, 164.**Phenylhydrazine-*p*-carboxylic Acid**  
(*p*-Hydrazinobenzoic acid).Needles or plates from  $H_2O$ . M.p.  $220-5^\circ$  decomp. Mod. sol. hot  $H_2O$ .Fischer, *Ann.*, 1882, 212, 337.Anchel, Schoenheimer, *J. Biol. Chem.*, 1936, 114, 543**Phenylhydrazine- $\beta$ -sulphonic Acid** $C_6H_5O_3N_2S$  MW, 188

Not known in free state.

 $\bullet NH_4$  salt: needles from EtOH. M.p.  $208^\circ$  decomp. Sol.  $H_2O$ . Mod. sol. EtOH. Insol.  $Et_2O$ ,  $C_6H_6$ .*Na* salt: leaflets from  $H_2O$ .Paal, Kretschmer, *Ber.*, 1894, 27, 1245.Bucherer, Schmidt, *J. prakt. Chem.*, 1909, 79, 402.**Phenylhydrazine-*m*-sulphonic Acid**  
(Hydrazinobenzene-*m*-sulphonic acid) $C_6H_5O_3N_2S$ 

MW, 188

Plates or needles +  $2H_2O$  from  $H_2O$ . Sol. 40 parts  $H_2O$  at  $22^\circ$ . Sol. hot  $H_2O$ . Spar. sol. EtOH,  $Et_2O$ .Limpricht, *Ber.*, 1888, 21, 3409.**Phenylhydrazine-*p*-sulphonic Acid**  
(Hydrazinobenzene-*p*-sulphonic acid).Needles or leaflets from  $H_2O$ . Sol. 200 parts  $H_2O$  at  $11^\circ$ , 30 parts  $H_2O$  at  $100^\circ$ . Spar. sol. EtOH.Thompson, *J. Soc. Dyers Colourists*, 1921, 37, 7. **$\alpha$ -Phenylhydrazinoacetic Acid** $C_8H_{10}O_2N_2$  MW, 166Leaflets from EtOH. M.p.  $168^\circ$  decomp. Spar. sol. EtOH. Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Sol. dil.  $HNO_3$  with red col. Reduces warm Fehling's.*B, HCl*: needles. M.p.  $170^\circ$  decomp.*Et ester*:  $C_{10}H_{14}O_2N_2$ . MW, 194. Oil. B.p.  $157-61^\circ/7$  mm. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin. Insol.  $H_2O$ . Reduces hot Fehling's.*Amide*:  $C_8H_{11}ON_3$ . MW, 165. Needles. M.p.  $150^\circ$ . Sol.  $C_6H_6$ .*Anilide*: needles from EtOH. M.p.  $149^\circ$ . Reduces Fehling's and  $NH_3 \cdot AgNO_3$  in the cold.Rupe, Heberlein, *Ann.*, 1898, 301, 58.Busch, *Ber.*, 1903, 36, 3880.Harries, *Ber.*, 1895, 28, 1225. **$\beta$ -Phenylhydrazinoacetic Acid** $C_8H_{10}O_2N_2$  MW, 166Yellow leaflets or needles from EtOH. M.p.  $153^\circ$  ( $173^\circ$  decomp.). Mod. sol. EtOH. Spar. sol.  $Me_2CO$ , AcOEt,  $C_6H_6$ .*B, HCl*: leaflets. M.p.  $165^\circ$  decomp.*Et ester*: oil. *Oxalate*: leaflets. M.p.  $156^\circ$ .*Phenyl ester*:  $C_{14}H_{14}O_2N_2$ . MW, 242. Leaflets. M.p.  $93-4^\circ$ .*Anilide*: m.p.  $135^\circ$ .

See first two references above and also

Busch, Meussdörfer, *J. prakt. Chem.*, 1907, 75, 124.Ghosh, Guha, *Chem. Zentr.*, 1934, I, 3050.**1- $\beta$ -Phenylhydrazinobutyric Acid** $C_{10}H_{14}O_2N_2$  MW, 194

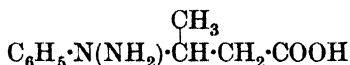
Needles from MeOH. Sublimes.

*Amide*:  $C_{10}H_{15}ON_3$ . MW, 193. Needles

from  $\text{Et}_2\text{O}$ -pet. ether. M.p.  $79^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Less sol.  $\text{C}_6\text{H}_6$ .

*Nitrile*:  $\text{C}_{10}\text{H}_{13}\text{N}_3$ . MW, 175. Cryst. from  $\text{Et}_2\text{O}$ -pet. ether. M.p.  $37^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ .

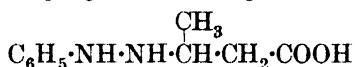
v. Miller, Plöchl, Sender, *Ber.*, 1892, **25**, 2037.

2- $\alpha$ -Phenylhydrazinobutyric Acid

$\text{C}_{10}\text{H}_{14}\text{O}_2\text{N}_2$  MW, 194

Plates from  $\text{EtOH}$ . M.p.  $111^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ .

Lederer, *J. prakt. Chem.*, 1891, **45**, 87.

2- $\beta$ -Phenylhydrazinobutyric Acid

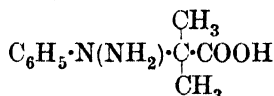
$\text{C}_{10}\text{H}_{14}\text{O}_2\text{N}_2$  MW, 194

Needles from  $\text{C}_6\text{H}_6$ . M.p.  $96-7^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol. ligroin.

Prentice, *J. Chem. Soc.*, 1904, **85**, 1671.

## Phenylhydrazinoformic Acid.

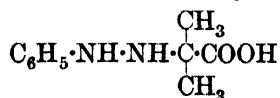
See Phenylhydrazine-carboxylic Acid.

1- $\alpha$ -Phenylhydrazinoisobutyric Acid

$\text{C}_{10}\text{H}_{14}\text{O}_2\text{N}_2$  MW, 194

*Amide*:  $\text{C}_{10}\text{H}_{15}\text{ON}_3$ . MW, 193. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $118^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ , hot  $\text{H}_2\text{O}$ . Spar. sol. ligroin. *B, HCl*: needles from  $\text{EtOH}$ . M.p.  $206^\circ$ .

v. Walther, Hübner, *J. prakt. Chem.*, 1916, **93**, 132.

1- $\beta$ -Phenylhydrazinoisobutyric Acid

$\text{C}_{10}\text{H}_{14}\text{O}_2\text{N}_2$  MW, 194

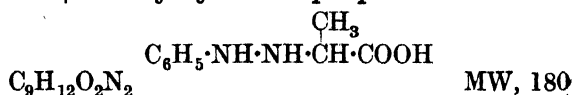
Leaflets from  $\text{EtOH}$ . M.p.  $165-6^\circ$ .

*Amide*: leaflets from  $\text{EtOH}$ . M.p.  $117^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin. Reduces Fehling's on heating.

*Nitrile*:  $\text{C}_{10}\text{H}_{13}\text{N}_3$ . MW, 175. Needles from ligroin. M.p.  $70^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin. Insol.  $\text{H}_2\text{O}$ . Reduces hot Fehling's.

Reissert, *Ber.*, 1884, **17**, 1461.

Eckstein, *Ber.*, 1892, **25**, 3323.

1- $\beta$ -Phenylhydrazinopropionic Acid

Needles from  $\text{MeOH}$ . M.p.  $174^\circ$ .

*Et ester*:  $\text{C}_{11}\text{H}_{16}\text{O}_2\text{N}_2$ . MW, 208. Liq. Very sol. dil.  $\text{HCl}$ . Reduces Fehling's in the cold.

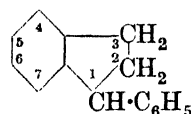
*Amide*:  $\text{C}_9\text{H}_{13}\text{ON}_3$ . MW, 179. Cryst. from  $\text{H}_2\text{O}$ . M.p.  $124^\circ$ . Sol.  $\text{EtOH}$ , hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin.

*Nitrile*:  $\text{C}_9\text{H}_{11}\text{N}_3$ . MW, 161. Cryst. from ligroin. M.p.  $58^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Less sol.  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin. Insol.  $\text{H}_2\text{O}$ . Reduces hot Fehling's. Fuming  $\text{HCl} \rightarrow$  amide.

Reissert, *Ber.*, 1884, **17**, 1453; 1892, **25**, 2701.

v. Miller, Plöchl, Rohde, *Ber.*, 1892, **25**, 2060.

## 1-Phenylhydrindene



$\text{C}_{15}\text{H}_{14}$  MW, 194

B.p.  $148-50^\circ/13$  mm.

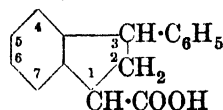
Mayer, Stieglitz, *Ber.*, 1921, **54**, 1399.

## 2-Phenylhydrindene.

Yellow oil. B.p.  $162-3^\circ/10$  mm.  $D_4^{16}$  1.0821.  $n_D^{15}$  1.5955.

v. Braun, Manz, *Ber.*, 1929, **62**, 1062.

## 3-Phenyl-1-hydrindenic Acid (3-Phenylhydrindene-1-carboxylic acid)



$\text{C}_{16}\text{H}_{14}\text{O}_2$  MW, 238

Needles from  $\text{AcOH}$ . M.p.  $143-144.5^\circ$ . B.p.  $227-9^\circ/14$  mm.

*Me ester*:  $\text{C}_{17}\text{H}_{16}\text{O}_2$ . MW, 252. Needles from  $\text{EtOH}$ -pet. ether. M.p.  $77-8^\circ$ . B.p.  $204.5-205^\circ/13$  mm.

*Et ester*:  $\text{C}_{18}\text{H}_{18}\text{O}_2$ . MW, 266. Prisms from  $\text{EtOH}$ . M.p.  $69-70^\circ$ .

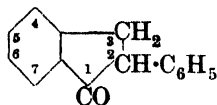
Blum-Bergmann, *Ann.*, 1930, **484**, 38; 1932, **492**, 277.

## 2-Phenyl-2-hydrindenic Acid (2-Phenylhydrindene-2-carboxylic acid).

Cryst. M.p.  $194-5^\circ$ .

Case, *J. Am. Chem. Soc.*, 1934, **56**, 715.

## 2-Phenyl-1-hydrindone (2-Phenylindanone)

 $C_{15}H_{12}O$  MW, 208

Prisms from EtOH. M.p. 77–8°. B.p. 344° decomp. Very sol.  $CHCl_3$ ,  $Me_2CO$ ,  $C_6H_6$ . Sol. EtOH,  $Et_2O$ . Spar. sol. ligroin. Reduces warm  $NH_3$ ,  $AgNO_3$ .

Semicarbazone: cryst. from MeOH. M.p. 211–12°.

Phenylhydrazone: cryst. from  $C_6H_6$ . M.p. 137–8°.

p-Nitrophenylhydrazone: yellow leaflets from  $Me_2CO$ , red cryst. powder from AcOH. M.p. 174°.

v. Miller, Rohde, *Ber.*, 1892, 25, 2096.

Auwers, Auffenberg, *Ber.*, 1919, 52, 107.

## 3-Phenyl-1-hydrindone (3-Phenylindanone).

Prisms from MeOH. M.p. 78°. Very sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ .

Oxime: needles from EtOH.Aq. M.p. 141°.

Semicarbazone: m.p. 223–5°. Spar. sol. most solvents.

Phenylhydrazone: yellowish cryst. from EtOH. M.p. 130°.

p-Nitrophenylhydrazone: red cryst. from AcOH. M.p. 220–1°.

Auwers, Auffenberg, *Ber.*, 1919, 52, 110.

Liebermann, Hartmann, *Ber.*, 1892, 25, 2124.

Pfeiffer, de Waal, *Ann.*, 1935, 520, 189.

## 4-Phenyl-1-hydrindone (4-Phenylindanone).

Thick yellow oil. B.p. 200–5°/11 mm.

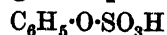
Semicarbazone: cryst. M.p. 192°. Very sol. EtOH.

v. Braun, Manz, *Ann.*, 1929, 468, 276.

## Phenylhydrocinnamic Acid.

See 1:2-, and 2:2-Diphenylpropionic Acids.

## Phenyl hydrogen sulphate

 $C_6H_6O_4S$  MW, 174

Very unstable.

Na salt: needles. Very hygroscopic. De-comp. easily.

K salt: leaflets from EtOH. Sol. 7 parts  $H_2O$  at 15°.

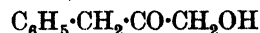
$NH_4$  salt: cryst. from dil.  $NH_3$ .Aq.  $FeCl_3 \rightarrow$  reddish-violet col.

Baumann, *Ber.*, 1878, 11, 1907.

Czapek, *Monatsh.*, 1914, 35, 639.

Hofmann, Biesalski, *Ber.*, 1912, 45, 1396.

## 3-Phenylhydroxyacetone (Phenylacetylcarbinol, phenacetylcarbinol, hydroxymethyl benzyl ketone, phenylacetol, 1-hydroxy-3-phenylacetone, phenylpropanolone)

 $C_9H_{10}O_2$  MW, 150

M.p. 48°. B.p. 144–5°/12–13 mm.  $Ag_2O \rightarrow$  phenylacetic acid.

Me ether:  $C_{10}H_{12}O_2$ . MW, 164. B.p. 139–40°/27 mm. Semicarbazone: m.p. 127–8°.

Et ether:  $C_{11}H_{14}O_2$ . MW, 178. B.p. 116–17°/1 mm. Semicarbazone: m.p. 100°.

Phenyl ether:  $C_{15}H_{14}O_2$ . MW, 226. Needles from ligroin. M.p. 43–4°. Semicarbazone: needles from MeOH. M.p. 151–2°. Phenylhydrazone: yellow cryst. from MeOH. Sinters at 65°, m.p. 94–6°.

Benzyl ether:  $C_{16}H_{16}O_2$ . MW, 240. B.p. 235°/40 mm. Semicarbazone: m.p. about 105°.

Oxime: m.p. 118°.

Semicarbazone: m.p. 134°.

Acetyl: needles from EtOH. M.p. 131°.

p-Nitrobenzoyl: plates from EtOH.Aq. M.p. 120°.

Darmon, *Compt. rend.*, 1933, 197, 1328, 1649.

 $\beta$ -Phenylhydroxylamine (N-Phenylhydroxylamine) $C_6H_7ON$  MW, 109

Needles from  $H_2O$ ,  $C_6H_6$  or pet. ether. M.p. 81–2°. Sol. 50 parts cold, 10 parts hot  $H_2O$ . Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ , hot  $C_6H_6$ . Spar. sol. ligroin. Conc.  $H_2SO_4 \rightarrow$  deep blue sol.

N-Formyl: plates or needles. M.p. 70–1°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , hot  $H_2O$ . Spar. sol. pet. ether.

N-Acetyl: needles from ligroin. M.p. 67–67.5°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , hot  $H_2O$ . Spar. sol. pet. ether.

O:N-Diacetyl: prisms from  $C_6H_6$ -pet. ether. M.p. 43°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ , boiling ligroin. Spar. sol.  $H_2O$ .

N-Benzoyl: needles from pet. ether. M.p. 123–4°. Spar. sol.  $Et_2O$ .

O:N-Dibenzoyl: cryst. from EtOH. M.p. 121°. Sol. hot EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. pet. ether.

N-Carboethoxyl: prisms from ligroin. M.p. 47–5°. B.p. 160–3°/12 mm. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Spar. sol. ligroin.

N-Nitroso: phenylisonitramine, phenyl-nitrosohydroxylamine. Needles from ligroin. M.p. 58–9°. Sol. usual solvents. Spar. sol.  $H_2O$ .

$k = 5.7 \times 10^{-6}$  at  $0^\circ$ .  $NH_4$  salt: cupferron. Glistening white plates. Reagent for separation of Fe, Cu, titanium, vanadium, zirconium, uranium.

Wohl, *Ber.*, 1894, **27**, 1435.

Bamberger, Destraz, *Ber.*, 1902, **35**, 1884.

Ciamician, Silber, *Ber.*, 1905, **38**, 1183.

Kamm, *Organic Syntheses*, Collective Vol. I, 435.

Marvel, *ibid.*, 171.

### Phenyl-hydroxynaphthylmethane.

See Benzylnaphthol.

### Phenyl-hydroxyphenyl-acrylic Acid.

See Hydroxystilbene- $\alpha$ -carboxylic Acid.

### Phenyl hydroxyphenyl Diketone.

See Hydroxybenzil.

### Phenyl hydroxystyryl Ketone.

See 2-, 3-, and 4-Hydroxychalkones.

### Phenyl $\omega$ -hydroxy-*p*-tolyl Ketone.

See 4-Hydroxymethyl-benzophenone.

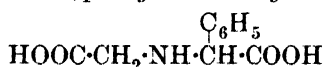
### Phenyliminazole.

See Phenylglyoxaline.

### Phenyliminodioxypbenzoin.

See Benzil-anil.

**1-Phenyliminodiacetic Acid** (*Phenyl- $\omega$ -carboxysarcosine, phenyl-N-carboxymethyl-glycine*)



$\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$  MW, 209

Needles from  $\text{H}_2\text{O}$ . M.p.  $220^\circ$  decomp. Spar. sol.  $\text{H}_2\text{O}$ . Insol. EtOH,  $\text{Et}_2\text{O}$ .

*B.HCl*: leaflets. Decomp. at  $220$ – $2^\circ$ .

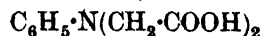
*Di-Me ester*:  $\text{C}_{12}\text{H}_{15}\text{O}_4\text{N}$ . MW, 237. Thick oil. B.p.  $220$ – $1^\circ/17$  mm.  $D_4^{16}$  1.1705,  $D_4^{25}$  1.1622.  $n_D^{18}$  1.5111.

*Di-Et ester*:  $\text{C}_{14}\text{H}_{19}\text{O}_4\text{N}$ . MW, 265. B.p.  $195$ – $6^\circ/17$  mm. Sol. EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .  $D_4^{20}$  1.1059.  $n_D^{20}$  1.4976.

*Diamide*:  $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}_3$ . MW, 207. Cryst. M.p.  $152$ – $3^\circ$ .

Stadnikow, *Ber.*, 1908, **41**, 4364; *Chem. Zentr.*, 1909, II, 1989.

**N-Phenyliminodiacetic Acid** (*Anil-diacetic acid, anilindiacetic acid*)



$\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$  MW, 209

Needles. M.p.  $152$ – $5^\circ$  decomp. Sol. EtOH, hot  $\text{H}_2\text{O}$ . Rather spar. sol.  $\text{Et}_2\text{O}$ .  $k = 2.73 \times 10^{-4}$  at  $25^\circ$ . Alc.  $\text{FeCl}_3 \rightarrow$  reddish-brown col.  $\rightarrow$  brown ppt. on standing.

*Di-Me ester*:  $\text{C}_{12}\text{H}_{15}\text{O}_4\text{N}$ . MW, 237. B.p.  $216$ – $18^\circ/25$  mm.,  $210$ – $11^\circ/18$  mm.

*Mono-Et ester*:  $\text{C}_{12}\text{H}_{15}\text{O}_4\text{N}$ . MW, 237.

Cryst. from  $\text{H}_2\text{O}$ . M.p.  $121$ – $2^\circ$ . B.p. about  $230$ – $40^\circ/17$  mm. *Anilide*: needles from  $\text{Et}_2\text{O}$ . M.p.  $121$ – $2^\circ$ .

*Di-Et ester*:  $\text{C}_{14}\text{H}_{19}\text{O}_4\text{N}$ . MW, 265. Oil. B.p.  $197$ – $8^\circ/17$  mm. ( $200$ – $6^\circ/6$  mm.).

*Diamide*:  $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}_3$ . MW, 207. Cryst. from  $\text{H}_2\text{O}$ . M.p.  $238^\circ$  ( $225^\circ$ ). Spar. sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

*Monoanilide*: needles from EtOH.Aq. M.p.  $215^\circ$ .

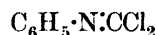
*Dianilide*: needles from EtOH. M.p.  $218^\circ$ .

Vorländer, Mumme, *Ber.*, 1901, **34**, 1647.

de Mouilpied, *J. Chem. Soc.*, 1905, **87**, 439.

Johnson, Bengis, *J. Am. Chem. Soc.*, 1911, **33**, 749.

**Phenyliminophosgene** (*Phenylcarbamide chloride*)



$\text{C}_7\text{H}_5\text{NCl}_2$  MW, 174

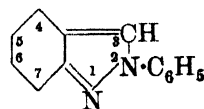
B.p.  $104$ – $6^\circ/30$  mm. Vapour is toxic and lachrymatory.

Bly, Perkins, Lewis, *J. Am. Chem. Soc.*, 1922, **44**, 2896.

### Phenyliminopropionic Acid.

See under Pyruvic Acid.

### 2-Phenylindazole



$\text{C}_{13}\text{H}_{10}\text{N}_2$  MW, 194

Needles from EtOH. M.p.  $83$ – $4^\circ$ . B.p.  $344$ – $5^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin. Insol.  $\text{H}_2\text{O}$ . Sol. conc. min. acids.  $\text{CrO}_3$  in AcOH, or  $\text{KMnO}_4 \rightarrow$  azobenzene-2-carboxylic acid.

$\text{B}_2\text{H}_2\text{PtCl}_6$ : m.p.  $187$ – $8^\circ$  decomp.

*Picrate*: yellow needles. M.p.  $93$ – $4^\circ$ .

Reissert, Lemmer, *Ber.*, 1926, **59**, 356.

Paal, *Ber.*, 1891, **24**, 961.

Busch, *Ber.*, 1894, **27**, 2899.

Freundler, *Bull. soc. chim.*, 1903, **29**, 745; 1904, **31**, 868.

### 3-Phenylindazole.

Exists in two forms

(i) Needles from ligroin. M.p.  $107$ – $8^\circ$ . Heated above m.p.  $\rightarrow$  (ii).

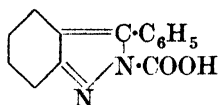
(ii) Prisms from ligroin. M.p.  $115$ – $16^\circ$ . Heated to higher temps. again  $\rightarrow$  (i).

1(?)*N-Acetyl*: needles from ligroin. M.p.  $69$ – $79^\circ$ .

Auwers, Schaum, *Ber.*, 1929, **62**, 1672.



## 3-Phenylindazole-2-carboxylic Acid

 $C_{14}H_{10}O_2N_2$ 

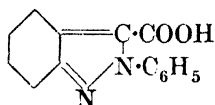
MW, 238

*Me ester*:  $C_{15}H_{12}O_2N_2$ . MW, 252. Needles from MeOH. M.p. 112–13°. Sol. EtOH, AcOH. Prac. insol.  $C_6H_6$ , pet. ether.

*Et ester*:  $C_{16}H_{14}O_2N_2$ . MW, 266. Needles. M.p. 83–4°.

Auwers, Hüttenes, *Ber.*, 1922, **55**, 1133.

## 2-Phenylindazole-3-carboxylic Acid

 $C_{14}H_{10}O_2N_2$ 

MW, 238

M.p. 200° decomp. Heat  $\rightarrow$  2-phenylindazole.

*Me ester*: needles from EtOH. M.p. 169–70°.

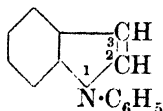
*Amide*:  $C_{14}H_{11}ON_3$ . MW, 237. M.p. 247–8° (243–4°).

*Nitrile*:  $C_{14}H_9N_3$ . MW, 219. M.p. 105° (106–7°).

Reissert, Lemmer, *Ber.*, 1926, **59**, 355.

Heller, Spielmeier, *Ber.*, 1925, **58**, 836.

## 1-Phenylindole (N-Phenylindole)

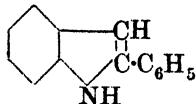
 $C_{14}H_{11}N$ 

MW, 193

Yellowish oil. B.p. 326–7°/757 mm. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ . Volatile in steam. With pine-splinter + HCl  $\rightarrow$  bluish-violet col.

Fischer, Hess, *Ber.*, 1884, **17**, 568.

Pfaff, *Ann.*, 1887, **239**, 221.

2-Phenylindole ( $\alpha$ -Phenylindole) $C_{14}H_{11}N$ 

MW, 193

Leaflets from EtOH or ligroin. M.p. 189° (187°). B.p. above 360°, about 250°/10 mm. Odour resembles indole. Gradually turns green

in air. Sublimes. Spar. volatile in steam. Sol.  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ , hot  $CS_2$ . Spar. sol. hot  $H_2O$ . Insol. dil. min. acids.  $CrO_3$  in boiling AcOH  $\rightarrow$  benzoic acid.

*N-Me*:  $C_{15}H_{13}N$ . MW, 207. Cryst. from EtOH,  $C_6H_6$ , or ligroin. M.p. 101°. Spar. volatile in steam. Mod. sol. cold conc. HCl, re-pptd. unchanged on dilution.

*N-Et*:  $C_{16}H_{15}N$ . MW, 221. Needles from EtOH. M.p. 86°.

*Picrate*: red prisms from EtOH. M.p. 127°.

I.C.I., E.P., 330,332, (*Chem. Abstracts*, 1930, **24**, 5770).

I.G., D.R.P., 574,840, (*Chem. Abstracts*, 1933, **27**, 4541).

Campbell, *J. Chem. Soc.*, 1935, 1210.

Wolff, *Ann.*, 1912, **394**, 107.

Madelung, *Ber.*, 1912, **45**, 1131.

Fischer, Hütz, *Ber.*, 1895, **28**, 587.

3-Phenylindole ( $\beta$ -Phenylindole).

Leaflets from ligroin. M.p. 88–9°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Mod. sol. hot ligroin. Insol.  $H_2O$ .  $ZnCl_2$  at 170°  $\rightarrow$  2-phenylindole.

*N-Me*: cryst. from pet. ether. M.p. 64–5°.

Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .  $ZnCl_2$  at 210°  $\rightarrow$

*N-methyl-2-phenylindole*. *Picrate*: brown needles from  $C_6H_6$ -ligroin. M.p. 90°.

*N-Nitroso*: yellow needles from ligroin. M.p. 60–1° Sol.  $Et_2O$ ,  $CHCl_3$ ,  $Me_2CO$ ,  $C_6H_6$ .

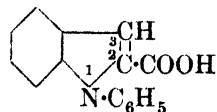
*Picrate*: red needles from  $C_6H_6$ -ligroin. M.p. 107°.

Fischer, Schmidt, *Ber.*, 1888, **21**, 1073, 1811.

Henle, *Ber.*, 1905, **38**, 1365.

Ince, *Ann.*, 1889, **253**, 38.

## 1-Phenylindole-2-carboxylic Acid

 $C_{15}H_{11}O_2N$ 

MW, 237

Needles from EtOH.Aq. M.p. 176°. Sol. EtOH,  $Et_2O$ . Very spar. sol. boiling  $H_2O$ . At 200–10°  $\rightarrow$  1-phenylindole.

Fischer, Hess, *Ber.*, 1884, **17**, 567.

Pfaff, *Ann.*, 1887, **239**, 221.

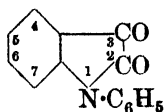
## 3-Phenylindole-2-carboxylic Acid.

Needles from  $C_6H_6$ . M.p. 186°.

*Et ester*:  $C_{17}H_{15}O_2N$ . MW, 265. Cryst. from EtOH. M.p. 137–8°.

Manske, Perkin, Robinson, *J. Chem. Soc.*, 1927, 6.

## N-Phenylisatin



$C_{14}H_9O_2N$  MW, 223

Yellowish-red leaflets from EtOH. M.p. 138°. Mod. sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

3-Hydrazone: yellow needles from EtOH. M.p. 192°.

Stollé, *Ber.*, 1913, **46**, 3915; D.R.P., 281,046, (*Chem. Zentr.*, 1915, I, 71).

Pföhl, *Ann.*, 1887, **239**, 222.

## 1-Phenylisoamyl Alcohol.

Isobutylphenylcarbinol, *q.v.*

2-Phenylisoamyl Alcohol (2-Methyl-3-phenylbutanol-4, 3-methyl-2-phenyl-n-butyl alcohol)

$C_{11}H_{16}O$  (CH<sub>3</sub>)<sub>2</sub>CH·CH(C<sub>6</sub>H<sub>5</sub>)·CH<sub>2</sub>OH MW, 164

Thick oil. B.p. 130°/15 mm.

Acetyl: liq. B.p. 134°/15 mm.

Ramart-Lucas, Amagat, *Compt. rend.*, 1927, **184**, 30.

Blaise, Courtot, *Bull. soc. chim.*, 1906, **35**, 595.

## N-Phenylisoamylamine.

See Isoamylaniline.

## 1-Phenylisobutane.

See Isobutylbenzene.

## 2-Phenylisobutane.

tert.-Butylbenzene, *q.v.*

## 2-Phenylisobutyl Alcohol

$C_{10}H_{14}O$  (CH<sub>3</sub>)<sub>2</sub>CH·CH<sub>2</sub>·CH(C<sub>6</sub>H<sub>5</sub>)·CH<sub>2</sub>OH MW, 150

B.p. 122–3°/20 mm.

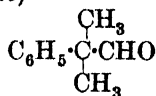
Phenylurethane: m.p. 59–60°.

Haller, Bauer, *Ann. chim.*, 1918, **9**, 10.

## Phenylisobutylamine.

See Aminoisobutylbenzene and N-Isobutylaniline.

1-Phenylisobutyraldehyde (Dimethyl-phenylacetaldehyde)



$C_{10}H_{12}O$  MW, 148

B.p. 215–18°, 105–10°/14 mm. D<sub>20</sub> 0.9912. Does not form bisulphite comp.

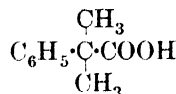
Semicarbazone: m.p. 172°.

Tiffeneau, Dorlencourt, *Ann. chim. phys.*, 1907, **10**, 366; 1909, **16**, 248.

## 2-Phenylisobutyraldehyde.

See α-Methylhydrocinnamaldehyde.

1-Phenylisobutyric Acid (1-Methylhydrotropic acid, dimethylphenylacetic acid)



$C_{10}H_{12}O_2$  MW, 164

Prisms from EtOH. M.p. 80–1°. B.p. 150–5°/10 mm. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. with green fluor.

Me ester: C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>. MW, 178. B.p. 225°.

Et ester: C<sub>12</sub>H<sub>16</sub>O<sub>2</sub>. MW, 192. B.p. 235–6°.

Propyl ester: C<sub>13</sub>H<sub>18</sub>O<sub>2</sub>. MW, 206. B.p. 250°.

Isobutyl ester: C<sub>14</sub>H<sub>20</sub>O<sub>2</sub>. MW, 220. B.p. 260–1°.

Chloride: C<sub>10</sub>H<sub>11</sub>OCl. MW, 182.5. B.p. 109°/13 mm.

Amide: C<sub>10</sub>H<sub>13</sub>ON. MW, 163. Cryst. M.p. 160–1°. B.p. 200–5°/60 mm. Spar. sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Nitrile: C<sub>10</sub>H<sub>11</sub>N. MW, 145. B.p. 232°, 114–16°/20 mm. D<sub>21</sub> 0.966. n<sub>D</sub> 1.50665.

Wallach, *Chem. Zentr.*, 1899, II, 1047.

Haller, Bauer, *Compt. rend.*, 1912, **155**, 1582.

## 2-Phenylisobutyric Acid.

See α-Methylhydrocinnamic Acid.

1-Phenylisocaproic Acid (3-Methyl-1-phenylvaleric acid, isobutylphenylacetic acid)

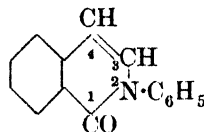
$C_{12}H_{16}O_2$  (CH<sub>3</sub>)<sub>2</sub>CH·CH<sub>2</sub>·CH(C<sub>6</sub>H<sub>5</sub>)·COOH MW, 192

Prisms from pet. ether. M.p. 78–9°.

Nitrile: C<sub>12</sub>H<sub>15</sub>N. MW, 173. B.p. 263–6°, 136–8°/15 mm. D<sub>16</sub> 0.942. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, AcOH. Insol. H<sub>2</sub>O. Volatile in steam.

Bodroux, Taboury, *Bull. soc. chim.*, 1910, **7**, 668.

N-Phenylisocarbostyryl (2-Phenylisoquinoline)



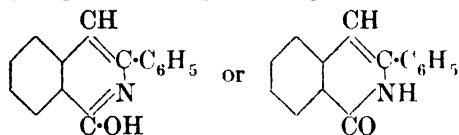
$C_{15}H_{11}ON$  MW, 221

Needles from EtOH.Aq. M.p. 117.5°. Very sol. EtOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Sublimes.

Bamberger, Frew, *Ber.*, 1894, **27**, 203.

Dieckmann, Meiser, *Ber.*, 1908, **41**, 3268.

**3-Phenylisocarbostyryl** (1-Hydroxy-3-phenylisoquinoline, 3-phenylisoquinolone)



C<sub>15</sub>H<sub>11</sub>ON MW, 221

Needles or prisms. M.p. 197°. Sol. hot EtOH, conc. HCl. Dist. with Zn dust in H → 3-phenylisoquinoline.

*Et ether*: C<sub>17</sub>H<sub>15</sub>ON. MW, 249. Needles from EtOH. M.p. 45-6°.

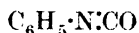
Gabriel, *Ber.*, 1886, **19**, 835.

Wölbling, *Ber.*, 1905, **38**, 3848.

**2-Phenylisocrotonic Acid.**

*Cis* form of β-Methylcinnamic Acid. See β-Methylcinnamic Acid.

**Phenyl isocyanate** (*Phenylcarbimide, carb-anil*)



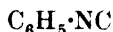
C<sub>7</sub>H<sub>5</sub>ON MW, 119

Pungent lachrymatory liq. B.p. 162-3°/751 mm., 55°/13 mm. D<sub>4</sub><sup>19.6</sup> 1.0956, D<sub>4</sub><sup>25.9</sup> 1.08870. n<sub>D</sub><sup>19.6</sup> 1.53684. Used for characterisation of alcohols and as a dehydrating agent.

Hardy, *J. Chem. Soc.*, 1934, 2011.

Zimmer, D.R.P., 133,760, (*Chem. Zentr.*, 1902, II, 553).

**Phenylisocyanide** (*Phenylcarbylamine, benzo-isonitrile*)

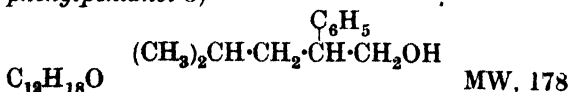


C<sub>7</sub>H<sub>5</sub>N MW, 103

Liq. with penetrating odour. B.p. 165-6° part. polymerisation, 78°/40 mm. D<sub>15</sub> 0.977. Very unstable. Rapidly turns green and then blue → slowly to a brown resin. Na + amyl alcohol → methylaniline. Heated with EtONa in sealed tube → N:N'-diphenylformamidine.

Biddle, Goldberg, *Ann.*, 1900, **310**, 7.

**1-Phenylisohexyl Alcohol** (2-Methyl-4-phenylpentanol-5)



C<sub>12</sub>H<sub>18</sub>O MW, 178

B.p. 138-9°/14 mm.

*Phenylurethane*: cryst. from Et<sub>2</sub>O-ligroin. M.p. 78°.

Ramart, Amagat, *Ann. chim.*, 1927, **8**, 288.

**Phenylisonitramine.**

See under Phenylhydroxylamine.

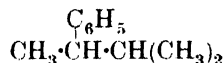
**Phenylisonitromethane.**

See under Phenylnitromethane.

**2-Phenylisopentane.**

*tert.*-Amylbenzene, *q.v.*

**3-Phenylisopentane** (2-Methyl-3-phenylbutane)



C<sub>11</sub>H<sub>16</sub> MW, 148

B.p. 188-9°/753 mm. D<sub>4</sub><sup>16</sup> 0.8672. n<sub>D</sub><sup>16</sup> 1.4972.

Konowalow, Jegorow, *Chem. Zentr.*, 1899, I, 776.

Klages, *Ber.*, 1903, **36**, 3691.

**4-Phenylisopentane.**

See Isoamylbenzene.

**5-Phenylisophthalic Acid.**

See Diphenyl-3:5-dicarboxylic Acid.

**Phenylisopropyl Alcohol.**

See Dimethylphenylcarbinol and Methylbenzylcarbinol.

**N-Phenylisopropylamine.**

*N*-Isopropylaniline, *q.v.*

**1-Phenylisopropylamine** (α-Aminocumene, 2-amino-2-phenylpropane, α-aminoisopropylbenzene)



C<sub>9</sub>H<sub>13</sub>N MW, 135

B.p. 196-7°/762 mm. D<sub>20</sub> 0.9424. n<sub>D</sub><sup>25</sup> 1.5181.

*B.HCl*: m.p. 235.5°. Sol. EtOH. Very spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. pet. ether.

*B.HNO<sub>2</sub>*: m.p. 98-9° decomp.

*N-Benzoyl*: needles from EtOH. M.p. 159°. Very spar. sol. Et<sub>2</sub>O.

*Oxalate*: needles from EtOH. M.p. 131°.

Konowalow, *Chem. Zentr.*, 1894, II, 33.

Brander, *Rec. trav. chim.*, 1918, **37**, 68.

**2-Phenylisopropylamine** (2-Amino-1-phenylpropane, 1-benzylethylamine, β-amino-propylbenzene)



C<sub>9</sub>H<sub>13</sub>N MW, 135

*d.*

B.p. 102°/16 mm. D<sub>4</sub><sup>15</sup> 0.949. n<sub>D</sub><sup>30</sup> 1.4704. [α]<sub>D</sub><sup>15</sup> + 35.6°.

*B.HCl*: m.p. 156°. [α]<sub>D</sub><sup>15</sup> + 24.8° in H<sub>2</sub>O.

*N-Benzoyl*: cryst. from EtOH. M.p. 159-60°. [α]<sub>D</sub><sup>15</sup> - 14.8° in C<sub>6</sub>H<sub>6</sub>.

*dl.*

B.p. 205° (203°).

*B, HCl*: m.p. 145–7°. Hygroscopic.

*N-Acetyl*: needles from EtOH.Aq. M.p. 64° initially, 93° on standing.

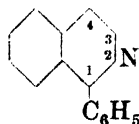
*Picrate*: m.p. 143°.

Hey, *J. Chem. Soc.*, 1930, 18.

Jones, Wallis, *J. Am. Chem. Soc.*, 1926, 48, 180.

Leithe, *Ber.*, 1932, 65, 664.

### 1-Phenylisoquinoline



$C_{15}H_{11}N$

MW, 205

Needles from EtOH.Aq. M.p. 95–6°. B.p. 298°/729 mm., 120–40°/1 mm. Spar. volatile in steam.

*B, HCl*: needles from EtOH–Et<sub>2</sub>O. M.p. 235–6°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellowish-red prisms from EtOH. M.p. 242–3° decomp.

*Picrate*: yellow needles from EtOH. M.p. 165°.

Pictet, Gams, *Ber.*, 1910, 43, 2388.

Späth, Berger, Kuntara, *Ber.*, 1930, 63, 139.

Rosenmund, Nothnagel, Riesenfeldt, *Ber.*, 1927, 60, 395.

### 3-Phenylisoquinoline.

Leaflets from EtOH. M.p. 103–5°. Spar. volatile in steam.

*B, HAuCl<sub>4</sub>, H<sub>2</sub>O*: m.p. 179–80°.

Gabriel, Neumann, *Ber.*, 1892, 25, 3573.

### Phenylisoquinolone.

See Phenylisocarbostyryl.

### 2-Phenylisoserine.

See 1-Hydroxy-2-amino-2-phenylpropionic Acid.

### 1-Phenylisosuccinic Acid.

See Methylphenylmalonic Acid.

### 2-Phenylisosuccinic Acid.

See Benzylmalonic Acid.

### Phenyl isothiocyanate

$C_6H_5 \cdot N \cdot CS$

$C_7H_5NS$

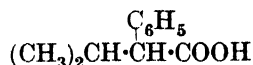
MW, 135

Colourless liq. F.P. – 21°. B.p. 221°, 131·8°/63 mm., 120–1°/35 mm., 95°/12 mm. *D*<sub>4</sub><sup>20</sup> 1·1477, *D*<sub>4</sub><sup>25</sup> 1·1288, *D*<sub>4</sub><sup>30</sup> 1·1202, *D*<sub>4</sub><sup>35</sup> 1·1061. *n*<sub>D</sub><sup>25</sup>

1·64918. Heat of comb. *C<sub>v</sub>* 1019·0 Cal., *C<sub>p</sub>* 1020·3 Cal.

Dains, Brewster, Olander, *Organic Syntheses*, Collective Vol. I, 437.

### 1-Phenylisovaleric Acid (*Isopropylphenylacetic acid*)



$C_{11}H_{14}O_2$

MW, 178

Prisms from ligroin. M.p. 61–2°. B.p. 159–60°/14 mm.

*Amide*:  $C_{11}H_{15}ON$ . MW, 177. Needles from EtOH.Aq. M.p. 111–12° (68°).

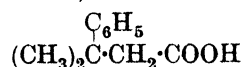
*Nitrile*:  $C_{11}H_{13}N$ . MW, 159. B.p. 245–9°/765 mm. *D*<sub>4</sub><sup>25</sup> 0·967. Sol. EtOH,  $C_6H_6$ . Insol.  $H_2O$ . Volatile in steam.

*Anilide*: m.p. 132–3°.

Bodroux, Taboury, *Bull. soc. chim.*, 1910, 7, 669.

Haller, Bauer, *Compt. rend.*, 1909, 149, 9. Hoffmann, *J. Am. Chem. Soc.*, 1929, 51, 2546.

### 2-Phenylisovaleric Acid ( $\beta$ : $\beta$ -*Dimethylhydrocinnamic acid*)



$C_{11}H_{14}O_2$

MW, 178

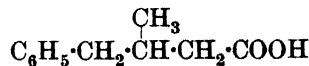
Cryst. from pet. ether. M.p. 58–58·5°.

*Me ester*:  $C_{12}H_{16}O_2$ . MW, 192. B.p. 120°/11 mm.

*Anilide*: cryst. from 60% MeOH. M.p. 122–3°.

Hoffmann, *J. Am. Chem. Soc.*, 1929, 51, 2545–6.

### 3-Phenylisovaleric Acid (*2-Benzylbutyric acid*)



$C_{11}H_{14}O_2$

MW, 178

Oil. B.p. 172°/13 mm. (161°/13 mm.). Sol. EtOH, Et<sub>2</sub>O. Insol.  $H_2O$ .

*Et ester*:  $C_{13}H_{18}O_2$ . MW, 206. B.p. 144–6°/15 mm.

*Chloride*:  $C_{11}H_{13}OCl$ . MW, 196·5. B.p. 133–5°/14 mm., 123°/12 mm.

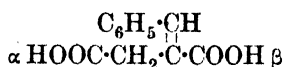
*Nitrile*:  $C_{11}H_{13}N$ . MW, 159. B.p. 121°/13 mm.

*Anilide*: needles from EtOH. M.p. 101°.

Anschütz, Motschmann, *Ann.*, 1915, 407, 88.

v. Braun, Stuckenschmidt, *Ber.*, 1923, 56, 1728.

**Phenylitaconic Acid** (*Benzylidenesuccinic acid*,  $\gamma$ -phenylitaconic acid)



$\text{C}_{11}\text{H}_{10}\text{O}_4$  MW, 206

Plates from  $\text{Et}_2\text{O}$ . M.p.  $192^\circ$  (rapid heat.) with loss of  $\text{H}_2\text{O}$ . Sol. 24 parts boiling  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , cold  $\text{H}_2\text{O}$ . Very spar. sol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ .  $k = 1.37 \times 10^{-4}$  at  $25^\circ$ . Heat at  $180-5^\circ$  in vacuo  $\rightarrow$  phenylitaconic anhydride + a little phenylcitraconic anhydride.  $\text{KMnO}_4 \rightarrow$  benzaldehyde, malonic and oxalic acids.

*Di-Me ester*:  $\text{C}_{13}\text{H}_{14}\text{O}_4$ . MW, 234. Thick oil. B.p.  $186^\circ/19$  mm. Misc. in all proportions with  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin. Insol.  $\text{H}_2\text{O}$ . Volatile in steam.

$\alpha$ -*Et ester*:  $\text{C}_{13}\text{H}_{14}\text{O}_4$ . MW, 234. Needles from  $\text{H}_2\text{O}$  or pet. ether. M.p.  $76-9^\circ$ .

$\beta$ -*Et ester*: needles or plates from  $\text{CS}_2$ . M.p.  $72^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol. pet. ether,  $\text{CS}_2$ .

*Di-Et ester*:  $\text{C}_{15}\text{H}_{18}\text{O}_4$ . MW, 262. Oil. B.p.  $315^\circ$ ,  $195^\circ/20$  mm. Polymerises slowly in the dark, more rapidly in light.  $n_D^{20}$  1.5243.

*Anhydride*:  $\text{C}_{11}\text{H}_8\text{O}_3$ . MW, 188. Leaflets from  $\text{CHCl}_3$ . M.p.  $164-6^\circ \rightarrow$  phenylcitraconic anhydride with slight decomp. Sol. 9 parts boiling  $\text{C}_6\text{H}_6$ , 200 parts boiling  $\text{Et}_2\text{O}$ . Very spar. sol. pet. ether,  $\text{CS}_2$ .

Fittig, Brooke, *Ann.*, 1899, 305, 21.

Stobbe, *Ber.*, 1908, 41, 4353.

Hecht, *Monatsh.*, 1903, 24, 367.

#### Phenyl-J Acid.

See *N*-Phenyl-2-amino-5-naphthol-7-sulphonic Acid.

#### 1-Phenyl-lactic Acid.

See Atrolactic Acid.

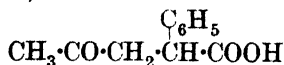
#### 2-Phenyl-lactic Acid.

See  $\alpha$ -Hydroxyhydrocinnamic Acid.

#### 2-Phenyl-lepidine.

See Flavoline.

**1-Phenyl-levulinic Acid** (*1-Phenyl-2-acetylpropionic acid*)



$\text{C}_{11}\text{H}_{12}\text{O}_3$  MW, 192

Leaflets from  $\text{H}_2\text{O}$ . M.p.  $126^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .

*Me ester*:  $\text{C}_{12}\text{H}_{14}\text{O}_3$ . MW, 206. M.p.  $71^\circ$ .

*Amide*:  $\text{C}_{11}\text{H}_{13}\text{O}_2\text{N}$ . MW, 191. Yellow prisms from  $\text{AcOH}$ . Aq. M.p.  $236^\circ$  decomp.

*Phenylhydrazone*: needles from  $\text{EtOH}$ . M.p.  $140^\circ$ .

Weltner, *Ber.*, 1885, 18, 790.

Ruhemann, *J. Chem. Soc.*, 1904, 85, 1455.

**4-Phenyl-levulinic Acid** (*2-Phenylacetylpropionic acid*)



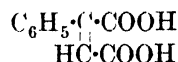
$\text{C}_{11}\text{H}_{12}\text{O}_3$  MW, 192

Needles from  $\text{CHCl}_3$ -ligroin. M.p.  $55-6^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{H}_2\text{O}$ . Spar. sol. ligroin.

*Semicarbazone*: needles from 50%  $\text{EtOH}$ . M.p.  $182-3^\circ$  decomp.

Stobbe, Russwurm, Schulz, *Ann.*, 1899, 308, 179.

**Phenylmaleic Acid** (*1-Phenylethylene-1:2-dicarboxylic acid*)



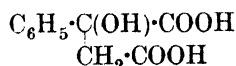
$\text{C}_{10}\text{H}_8\text{O}_4$  MW, 192

Prisms from  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$ . Very unstable.

*Anhydride*: needles from  $\text{CS}_2$ . M.p.  $119-119.5^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{CS}_2$ , ligroin. Cold  $\text{H}_2\text{O} \rightarrow$  acid.

Alexander, *Ann.*, 1890, 258, 77.

#### 1-Phenylmalic Acid

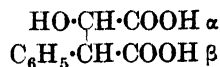


$\text{C}_{10}\text{H}_{10}\text{O}_5$  MW, 210

Prisms from  $\text{CHCl}_3$ . M.p.  $187-8^\circ$ . Sol. 75 parts  $\text{H}_2\text{O}$  at  $15^\circ$ . Sol.  $\text{Et}_2\text{O}$ .

Alexander, *Ann.*, 1890, 258, 76.

#### 2-Phenylmalic Acid



$\text{C}_{10}\text{H}_{10}\text{O}_5$  MW, 210

Cryst. from  $\text{Et}_2\text{O}$ . M.p.  $160^\circ$ . Sol. 3 parts  $\text{H}_2\text{O}$  at  $15^\circ$ . Insol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin.

$\beta$ -*Et ester*:  $\text{C}_{12}\text{H}_{14}\text{O}_5$ . MW, 238. Oil.  $\alpha$ -*Nitrile*:  $\text{C}_{12}\text{H}_{13}\text{O}_3\text{N}$ . MW, 219. Prisms from hot  $\text{EtOH}$ . M.p.  $127-8^\circ$ . Sol.  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ , ligroin.

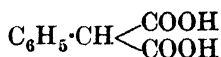
Alexander, *Ann.*, 1890, 258, 80.

Börner, *Chem. Zentr.*, 1900, I, 123.

#### Phenylmalonaldehydic Acid.

See Phenylformylacetic Acid.

## Phenylmalonic Acid

 $\text{C}_9\text{H}_8\text{O}_4$ 

MW, 180

Prisms from  $\text{H}_2\text{O}$ . M.p.  $152-3^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Less sol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ , ligroin. Above m.p.  $\rightarrow$  phenylacetic acid.

*Me ester*:  $\text{C}_{10}\text{H}_{10}\text{O}_4$ . MW, 194. Cryst. from  $\text{MeOH}$ . M.p.  $95^\circ$ . *Anilide*: m.p.  $109^\circ$ .

*Di-Me ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_4$ . MW, 208. Cryst. from ligroin. M.p.  $51^\circ$ . B.p.  $147-9^\circ/10$  mm.

*Et ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_4$ . MW, 208. *Amide*:  $\text{C}_{11}\text{H}_{13}\text{O}_3\text{N}$ . MW, 207. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $152^\circ$ . Sol.  $\text{EtOH}$ . Insol.  $\text{H}_2\text{O}$ .

*Di-Et ester*:  $\text{C}_{13}\text{H}_{16}\text{O}_4$ . MW, 236. B.p.  $171^\circ/21$  mm.,  $158-9^\circ/10$  mm.

*Dichloride*:  $\text{C}_9\text{H}_6\text{O}_2\text{Cl}_2$ . MW, 217. B.p.  $122^\circ/15$  mm.

*Diamide*:  $\text{C}_9\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 178. Cryst. from  $\text{EtOH}$ . M.p.  $233^\circ$ . Sol.  $\text{EtOH}$ . Insol.  $\text{H}_2\text{O}$ .

*Nitrile*: see Phenylcyanoacetic Acid.

*Dinitrile*:  $\text{C}_9\text{H}_6\text{N}_2$ . MW, 142. Cryst. from  $\text{EtOH}$ . Aq. M.p.  $68-9^\circ$ . B.p.  $152-3^\circ/21$  mm. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin.

Nelson, Cretcher, *J. Am. Chem. Soc.*, 1928, **50**, 2758.

Hessler, *Am. Chem. J.*, 1905, **32**, 123.

Wislicenus, *Ber.*, 1894, **27**, 1093.

Staudinger, Hirzel, *Ber.*, 1917, **50**, 1030.

Rising, Stieglitz, *J. Am. Chem. Soc.*, 1918, **40**, 728.

Dox, Yoder, *J. Am. Chem. Soc.*, 1922, **44**, 1564.

Ivanoff, Spassoff, *Bull. soc. chim.*, 1931, **49**, 19.

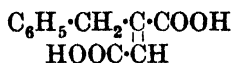
Blum-Bergmann, *Ber.*, 1932, **65**, 115.

## Phenyl Mercaptan.

See Thiophenol.

## Phenylmercaptobenzoic Acid.

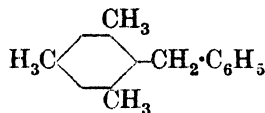
See Diphenyl sulphide carboxylic Acid.

Phenylmesaconic Acid (*Benzylfumaric acid*) $\text{C}_{11}\text{H}_{10}\text{O}_4$ 

MW, 206

Needles from  $\text{H}_2\text{O}$ . M.p.  $212^\circ$ . Sol.  $\text{Et}_2\text{O}$ , boiling  $\text{C}_6\text{H}_6$ . Sol. 35 parts boiling  $\text{H}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Insol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ , ligroin. Heat with 10%  $\text{NaOH} \rightarrow$  phenylitaconic acid + phenylatronic acid.

Fittig, Brooke, *Ann.*, 1899, **305**, 31.

Phenylmesitylmethane (*Benzylmesitylene*, 1 : 3 : 5-trimethyldiphenylmethane) $\text{C}_{16}\text{H}_{18}$ 

MW, 210

Needles. M.p.  $37^\circ$ . B.p.  $301-3^\circ$ ,  $183^\circ/11$  mm.  $\text{CrO}_3 \rightarrow$  benzoylmesitylene.  $\text{HI} + \text{P} \rightarrow$  toluene + mesitylene.

Louise, *Ann. chim. phys.*, 1885, **6**, 177.

Klages, Allendorff, *Ber.*, 1898, **31**, 1001.

Phenyl *p*-methoxyphenacyl Ether.

See under *p*-Hydroxyphenacyl Alcohol.

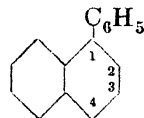
## Phenyl methoxystyryl Ketone.

See Anisylideneacetophenone and under 2-, and 3-Hydroxychalkones.

## Phenyl methylstyryl Ketone.

See Dypnone and 2'-, 3'-, and 4'-Methyl chalkones.

## 1-Phenylnaphthalene

 $\text{C}_{16}\text{H}_{12}$ 

MW, 204

Cryst. M.p. about  $45^\circ$ . B.p.  $334^\circ/760$  mm.,  $186-8^\circ/10$  mm. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ . Shows faint blue fluor.

Chattaway, *J. Chem. Soc.*, 1893, **63**, 1185.

Möhlau, Berger, *Ber.*, 1893, **26**, 1198.

Vesely, Štursa, *Chem. Zentr.*, 1936, **I**, 1221.

## 2-Phenylnaphthalene.

Leaflets from  $\text{EtOH}$ . M.p.  $101-2^\circ$ . B.p.  $345-6^\circ$ . Sol.  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ . Less sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Volatile in steam. Shows weak blue fluor.

Smith, Takamatsu, *J. Chem. Soc.*, 1881, **39**, 547.

Späth, *Monatsh.*, 1912, **33**, 1046.

## Phenylnaphthalene-carboxylic Acid.

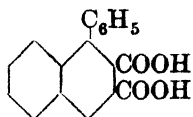
See  $\beta$ -Chrysenic Acid and Phenylnaphthoic Acid.

## 2-Phenylnaphthalene-1 : 2'-dicarboxylic Acid.

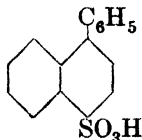
See Chrysodiphenic Acid.

**1-Phenylnaphthalene-2 : 3-dicarboxylic Acid**

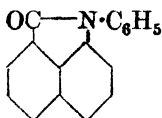
480

**N-Phenyl-1-naphthylamine****1-Phenylnaphthalene-2 : 3-dicarboxylic Acid** $C_{18}H_{12}O_4$ 

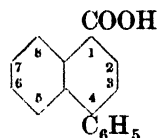
MW, 292

Leaflets from  $H_2O$ . At  $255^\circ \rightarrow$  anhydride.2-*Me ester*:  $C_{19}H_{14}O_4$ . MW, 306. Needles from EtOH or  $C_6H_6$ -ligroin. M.p.  $207^\circ$  decomp.Di-*Me ester*:  $C_{20}H_{16}O_4$ . MW, 320. Leaflets from MeOH.Aq. M.p.  $118-20^\circ$  ( $121^\circ$ ). Sol. hot EtOH. Spar. sol. Et<sub>2</sub>O.2-*Et ester*:  $C_{20}H_{16}O_4$ . MW, 320. Needles from  $C_6H_6$ -ligroin. M.p.  $207^\circ$  decomp. Sol. most org. solvents. Insol.  $H_2O$ .Di-*Et ester*:  $C_{22}H_{20}O_4$ . MW, 348. Plates from ligroin. M.p.  $129-30^\circ$ .Anhydride: needles from  $C_6H_6$ -ligroin. M.p.  $255^\circ$  ( $257-9^\circ$ ). Sol.  $CHCl_3$ , AcOH,  $C_6H_6$ . Spar. sol. EtOH. Insol. ligroin.Imide: needles from EtOH. M.p.  $246^\circ$ . Sol. EtOH, AcOH,  $C_6H_6$ .Lanser, Halvorsen, *Ber.*, 1902, **35**, 1407.Stobbe, *Ber.*, 1907, **40**, 3373.Michael, Bucher, *Am. Chem. J.*, 1898, **20**, 95.Pfeiffer, Moller, *Ber.*, 1907, **40**, 3841.**1-Phenylnaphthalene-4-sulphonic Acid** $C_{16}H_{12}O_3S$ 

MW, 284

Needles from  $C_6H_6$ . M.p.  $165-7^\circ$ .Anilide: cryst. from  $C_6H_6$ -pet. ether. M.p.  $167^\circ$ . Very sol.  $C_6H_6$ .v. Braun, Anton, *Ber.*, 1934, **67**, 1053.**N-Phenylnaphthastyril** $C_{17}H_{11}ON$ 

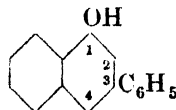
MW, 245

Yellow needles from ligroin. M.p.  $104-5^\circ$ . Mod. sol. usual org. solvents, hot conc. HCl. Sol. hot, insol. cold NaOH.Aq.I.G., F.P., 778,254, (*Chem. Abstracts*, 1935, **29**, 4600).Rule, Turner, *J. Chem. Soc.*, 1935, 318.**2-Phenyl-1-naphthoic Acid.**See  $\beta$ -Chrysenic Acid.**4-Phenyl-1-naphthoic Acid (4-Phenylnaphthalene-1-carboxylic acid)** $C_{17}H_{12}O_2$ 

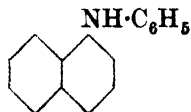
MW, 248

Cryst. from AcOH.Aq. M.p.  $172-3^\circ$ .Nitrile:  $C_{17}H_{11}N$ . MW, 229. Cryst. from  $C_6H_6$ . M.p.  $114-15^\circ$ .v. Braun, Anton, *Ber.*, 1934, **67**, 1054.**5-Phenyl-1-naphthoic Acid (5-Phenylnaphthalene-1-carboxylic acid).**Cryst. from AcOH. M.p.  $229^\circ$ .Nitrile: cryst. from  $C_6H_6$ . M.p.  $73-7^\circ$ .

See previous reference.

**3-Phenyl-1-naphthol (4-Hydroxy-2-phenylnaphthalene)** $C_{16}H_{12}O$ 

MW, 220

Brownish needles from EtOH.Aq. M.p.  $100-1^\circ$ . Sol. EtOH, Et<sub>2</sub>O,  $CHCl_3$ . Spar. sol. boiling  $H_2O$ .Ruhemann, *J. Chem. Soc.*, 1910, **97**, 461.**4-Phenyl-1-naphthol (4-Hydroxy-1-phenylnaphthalene).**Needles from  $C_6H_6$ -pet. ether. M.p.  $140^\circ$ .v. Braun, Anton, *Ber.*, 1934, **67**, 1053.**N-Phenyl-1-naphthylamine** $C_{16}H_{13}N$ 

MW, 219

Prisms or needles from EtOH, leaflets from ligroin. M.p.  $62^\circ$ . B.p.  $335^\circ/528$  mm.,  $226^\circ/8$  mm. ( $200^\circ/10$  mm.). Sol. EtOH, Et<sub>2</sub>O,  $CHCl_3$ , AcOH,  $C_6H_6$ . Sols. show blue fluor. Heat of comb.  $C_v$  2017 Cal. Conc.  $H_2SO_4 + HNO_3 \rightarrow$  blue col.  $\rightarrow$  green  $\rightarrow$  brown on heating. Rubber vulcanisation acceleration.N-Acetyl: cryst. from EtOH. M.p.  $115^\circ$ . Sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. Et<sub>2</sub>O.

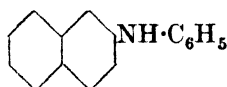
N-Benzoyl: cryst. from EtOH. M.p. 152°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Knoevenagel, *J. prakt. Chem.*, 1914, **89**, 17.

Knoll, D.R.P., 241,853, (*Chem. Zentr.*, 1912, I, 178).

Streiff, *Ann.*, 1881, **209**, 154.

#### N-Phenyl-2-naphthylamine



C<sub>16</sub>H<sub>13</sub>N

MW, 219

Needles from MeOH. M.p. 108° (102-4°). B.p. 395-399.5°, 237°/13 mm. Mod. sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>. Sols. show blue fluor. Heat of comb. C<sub>v</sub> 2007 Cal. Rubber vulcanisation accelerator.

N-Acetyl: cryst. from Et<sub>2</sub>O. M.p. 93°. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

N-Benzoyl: needles from EtOH. M.p. 136° (147-8°). Sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH.

N-Me: C<sub>17</sub>H<sub>15</sub>N. MW, 233. M.p. 52-3°.

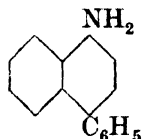
N-Et: C<sub>18</sub>H<sub>17</sub>N. MW, 247. Leaflets. M.p. 58°.

Streiff, *Ann.*, 1881, **209**, 157.

Cherntzov, Drozdov, *Chem. Abstracts*, 1935, **29**, 2529.

See also first two references above.

#### 4-Phenyl-1-naphthylamine



C<sub>16</sub>H<sub>13</sub>N

MW, 219

Cryst. M.p. 73-4°.

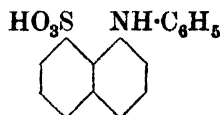
N-Acetyl: m.p. 167-8°.

Vesely, Štursa, *Chem. Zentr.*, 1933, II, 3850.

#### Phenylnaphthylamine-carboxylic Acid.

See Naphthylanthranilic Acid.

N-Phenyl-1-naphthylamine-8-sulphonic Acid (Phenyl-peri acid, 1-anilinonaphthalene-8-sulphonic acid)



C<sub>16</sub>H<sub>18</sub>O<sub>3</sub>NS

MW, 299

Leaflets. Spar. sol. H<sub>2</sub>O. Intermediate for azo dyes.

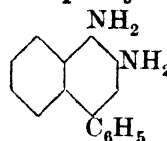
Na salt: needles + H<sub>2</sub>O. Very sol. H<sub>2</sub>O.

Bayer, D.R.P., 70,349.

Kalle, D.R.P., 170,630, (*Chem. Zentr.*, 1906, II, 473).

A.G.F.A., D.R.P., 158,923, (*Chem. Zentr.*, 1905, I, 909).

#### 4-Phenyl-1 : 2-naphthylenediamine



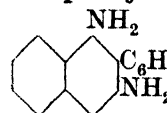
C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>

MW, 234

Cryst. M.p. 100-1°.

Vesely, Štursa, *Chem. Zentr.*, 1933, II, 3850.

#### 2-Phenyl-1 : 3-naphthylenediamine



C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>

MW, 234

Plates from MeOH. M.p. 116°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold Et<sub>2</sub>O. Insol. pet. ether. Turns red in air.

1 : 3-N-Di-Me: C<sub>18</sub>H<sub>18</sub>N<sub>2</sub>. MW, 262. *Cis*: Dimorphous. Needles or prisms from EtOH. M.p. 170°. Needles are stable between 20° and 80°. Prisms formed below 17° and above 80°. *Trans*: cryst. from EtOH. M.p. 159-60°. *Acetyl*: prisms from EtOH. M.p. 203°. *Di-acetyl*: prisms from MeOH.Aq. M.p. 207-8°. *p-Toluenesulphonyl*: needles from EtOH. M.p. 219-20°. *Di-p-toluenesulphonyl*: plates from Py. M.p. 305°.

N : N-Tri-Me: C<sub>19</sub>H<sub>20</sub>N<sub>2</sub>. MW, 276. Needles from EtOH. M.p. 104-5°. *Acetyl*: needles from EtOH. M.p. 178°.

N : N-Tetra-Me: C<sub>20</sub>H<sub>22</sub>N<sub>2</sub>. MW, 290. Prisms from EtOH. M.p. 122°.

3-N-Acetyl: plates from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 220°.

1 : 3-N-Diacetyl: needles. M.p. 267°.

1 : 3-N-Di-p-toluenesulphonyl: *cis*, dimorphous prisms from MeOH. M.ps. 203-5° and 188-9°. *Trans*: needles from MeOH. M.p. 173-5°.

N : N-Tri-p-toluenesulphonyl: needles from MeOH. M.p. 153-4°.

Atkinson, Thorpe, *J. Chem. Soc.*, 1906, **89**, 1934.

Atkinson, Ingham, Thorpe, *J. Chem. Soc.*, 1907, **91**, 589.

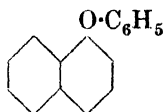
Lees, Thorpe, *ibid.*, 1296.

Gibson, Kentish, Simonsen, *J. Chem. Soc.*, 1928, 2131.

Lesslie, Turner, *J. Chem. Soc.*, 1929, 1512.



**Phenyl 1-naphthyl Ether** (1-Phenoxy-naphthalene)



$C_{16}H_{12}O$  MW, 220

Prisms from  $Et_2O$ -EtOH. M.p. 55-6°. B.p. 349.5°. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. EtOH, ligroin.

Ullmann, Sponagel, *Ann.*, 1906, 350, 90.  
Fritzsche, D.R.P., 269,543, (*Chem. Zentr.*, 1914, I, 591).

**Phenyl 2-naphthyl Ether** (2-Phenoxy-naphthalene).

Needles from  $Et_2O$ -EtOH. M.p. 46°. B.p. 335.5°/753 mm. Sol.  $Et_2O$ ,  $CHCl_3$ , AcOH. Insol.  $H_2O$ .

See previous references.

**sym.-Phenyl-1-naphthylhydrazine** (Benzenehydrazo- $\alpha$ -naphthalene)

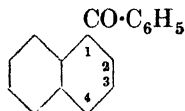
$C_6H_5 \cdot NH \cdot NH \cdot C_{10}H_7$  MW, 234

Leaflets. M.p. 125°.

Diacetyl: m.p. 264°.

Nietzki, Zehntner, *Ber.*, 1883, 26, 144.

**Phenyl 1-naphthyl Ketone** (1-Benzoyl-naphthalene)



$C_{17}H_{12}O$  MW, 232

Prisms from EtOH. M.p. 75.5-76°. B.p. 385°, 225°/12 mm., 222°/8 mm. Sol. 41 parts EtOH at 12°. Diamagnetic.

*Oxime*: exists in two forms. (i) Cryst. from EtOH. M.p. 161°. (ii) Cryst. from EtOH. M.p. 127°.

*Imide*: m.p. 68-9°. B.p. 181.5°/4.5 mm.

Moureu, Mignouac, *Compt. rend.*, 1913, 156, 1806.

Betti, Poccianti, *Gazz. chim. ital.*, 1915, 45, i, 374.

Caille, *Compt. rend.*, 1911, 153, 393.

Szperl, *Chem. Zentr.*, 1930, I, 3436.

**Phenyl 2-naphthyl Ketone** (2-Benzoyl-naphthalene).

Needles from EtOH. M.p. 82°. B.p. 398°/754 mm., 225°/8 mm. Sol. 49 parts EtOH at 12°. Paramagnetic.

*Oxime*: exists in two forms. (i) Needles from EtOH. M.p. 174°. (ii) Cryst. from EtOH. M.p. 157°.

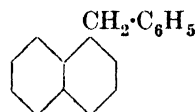
*Semicarbazone*: cryst. from EtOH. M.p. 175°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Insol.  $H_2O$ .

Lecher, *Ber.*, 1913, 46, 2667; D.R.P., 281,802, (*Chem. Zentr.*, 1915, I, 281).

Poccianti, *Gazz. chim. ital.*, 1915, 45, ii, 114.

Barbot, *Bull. soc. chim.*, 1930, 47, 1314.

**Phenyl-1-naphthylmethane** (1-Benzyl-naphthalene)



$C_{17}H_{14}$  MW, 218

M.p. 58.5°. B.p. 350°, 217-20°/20 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ .  $D^{17}$  1.166. Dil.  $HNO_3 \rightarrow$  phenyl 1-naphthyl ketone.

*Picrate*: yellow needles. M.p. 100-1°.

Nenitzescu, Isăcescu, Ionescu, *Ann.*, 1931, 491, 217.

Miquel, *Bull. soc. chim.*, 1876, 26, 2.

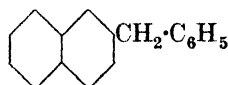
Roux, *Ann. chim. phys.*, 1887, 12, 326.

Lecher, D.R.P., 281,802, (*Chem. Zentr.*, 1915, I, 281).

Dziewoński, Dzicielewski, *Chem. Zentr.*, 1928, I, 57.

Gasopoulos, *Chem. Zentr.*, 1933, II, 43.

**Phenyl-2-naphthylmethane** (2-Benzyl-naphthalene)



$C_{17}H_{14}$  MW, 218

Prisms. M.p. 55.5°. B.p. 350°. Sol. EtOH,  $C_6H_6$ .  $D^0$  1.176. Dil.  $HNO_3 \rightarrow$  phenyl 2-naphthyl ketone.

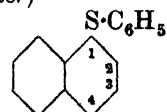
*Picrate*: golden-yellow needles from EtOH. M.p. 93°.

Roux, *Ann. chim. phys.*, 1887, 12, 326.

Vincent, Roux, *Bull. soc. chim.*, 1883, 40, 165.

Dziewoński, Wodelski, *Chem. Zentr.*, 1933, I, 774.

**Phenyl 1-naphthyl sulphide** (1-Thio-naphthol phenyl ether)



$C_{16}H_{12}S$

MW, 236

Prisms from EtOH.Aq. M.p. 41.8°. B.p. 255-6°/43 mm., 220-5°/11 mm.  $D_4^{25}$  1.167. Mod. sol. EtOH, Et<sub>2</sub>O.

Wuyts, *Bull. soc. chim.*, 1906, **35**, 167.

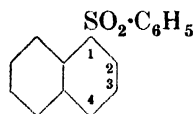
Bourgeois, *Ber.*, 1895, **28**, 2327.

**Phenyl 2-naphthyl sulphide** (2-Thio-naphthol phenyl ether).

Needles or leaflets from EtOH. M.p. 51.8°. B.p. 226°/11 mm.

See last reference above.

### Phenyl 1-naphthyl sulphone



$C_{16}H_{12}O_2S$

MW, 268

Cryst. from EtOH. M.p. 99.5-100.5°. Very sol. hot EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

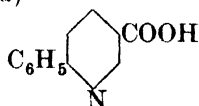
Michael, Adair, *Ber.*, 1877, **10**, 585.

### Phenyl 2-naphthyl sulphone.

Needles from EtOH or Et<sub>2</sub>O. M.p. 115-16°.

See previous reference.

### 6-Phenylnicotinic Acid (6-Phenylpyridine-3-carboxylic acid)



$C_{12}H_9O_2N$

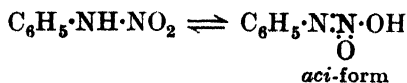
MW, 199

Needles from H<sub>2</sub>O. M.p. 232-3° (sinters at 220°). Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, AcOH.

Anilide: leaflets from EtOH. M.p. 199°.

Bemary, Paille, *Ber.*, 1924, **57**, 832.

### Phenylnitramine (N-Nitroaniline, nitraminobenzene)



$C_6H_5O_2N_2$

MW, 138

Pearly leaflets from pet. ether. M.p. 46-46.5°. Very sol. most org. solvents. Mod. sol. H<sub>2</sub>O. Spar. sol. pet. ether.  $k = 1.7 \times 10^{-5}$  at 18°,  $2.3 \times 10^{-5}$  at 25°. Explodes on strong heating. Warm dil. min. acids → o- and p-nitroaniline. Mod. volatile in steam.

N-Me: see Methylphenylnitramine.

aci-Me ether:  $C_7H_8O_2N_2$ . MW, 152. Yellow

Dict. of Org. Comp.—III.

oil with odour of heliotrope. Decomp. with explosion on warming.

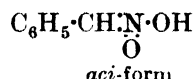
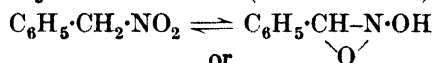
Bamberger, *Ber.*, 1893, **26**, 485; 1894, **27**, 362, 915.

Degner, v. Pechmann, *Ber.*, 1897, **30**, 647.

### Phenyl nitrobenzyl Ketone.

See Nitrodeoxybenzein.

### Phenylnitromethane (ω-Nitrotoluene)



$C_7H_7O_2N$

MW, 137

Yellow liq. B.p. 225-7°, 141-2°/35 mm., 118-19°/16 mm.  $D_4^{20}$  1.1756,  $D_4^{20}$  1.1598.  $n_D^{20}$  1.53230. Gives no col. with FeCl<sub>3</sub>. Alkalis → aci-form. Electrolytic reduction → benzylamine.

aci-Form: phenylisonitromethane. Cryst. from Et<sub>2</sub>O-pet. ether. M.p. 84°. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. C<sub>6</sub>H<sub>6</sub>. Insol. pet. ether. Sol. Na<sub>2</sub>CO<sub>3</sub>.Aq. FeCl<sub>3</sub> → intense reddish-brown col. NH<sub>4</sub> salt: powder. M.p. 89-90°.

Hantzsch, Schultze, *Ber.*, 1896, **29**, 700.

Konowalow, *Ber.*, 1895, **28**, 1861.

Holleman, *Rec. trav. chim.*, 1894, **13**, 405.

Wislicenus, Grützner, *Ber.*, 1909, **42**, 1932.

Schorygin, Ssokolowa, *Chem. Zentr.*, 1930, **II**, 2637.

### S-Phenyl-4-nitrothiosalicylic Acid.

See 5-Nitrodiphenylsulphide-2-carboxylic Acid.

### Phenyl 6-nitro-m-tolyl Ether.

See 6-Nitro-3-methyldiphenyl Ether.

### Phenyl nitrotolyl Ketone.

See Nitromethylbenzophenone.

### Phenyl nitrotolyl sulphide.

See Nitromethyldiphenyl sulphide.

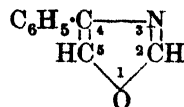
### Phenyl nitrotolyl sulphone.

See Nitromethyldiphenyl sulphone.

### Phenyloctane.

See Octylbenzene.

### 4-Phenyloxazole



$C_9H_7ON$

MW, 145

Thick liq. M.p. 6°. B.p. 220-2°.

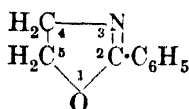
B,HCl: plates. M.p. 80°.

Lewy, *Ber.*, 1887, **20**, 2578.

Blümlein, *Ber.*, 1884, **17**, 2580.

**5-Phenyloxazole.**

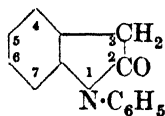
Cryst. M.p. 41-2°.

 $B_2, AuCl_3$ : orange needles from  $H_2O$ . M.p. 149-50°.Chloroplatinate: long yellow needles. Does not decompose below 275°. Spar. sol.  $H_2O$ .Bachstetz, *Ber.*, 1914, **47**, 3165.**2-Phenyl-2-oxazoline** (2-Phenyl-4:5-dihydro-oxazoline) $C_9H_9ON$ 

MW, 147

B.p. 246-8° (242-3°). Misc. with EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

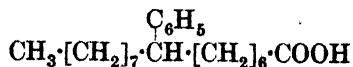
Picrate: yellow needles. M.p. 177°. Spar. sol. most solvents.

Gabriel, Neumann, *Ber.*, 1892, **25**, 2385.Gabriel, Stelzner, *Ber.*, 1895, **28**, 2933.Wenker, *J. Am. Chem. Soc.*, 1935, **57**, 1080.**1-Phenyloxindole** $C_{14}H_{11}ON$ 

MW, 209

Leaflets from  $H_2O$  or ligroin. M.p. 121°. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .Stollé, *Ber.*, 1914, **47**, 2120; D.R.P., 335,673, (*Chem. Abstracts*, 1923, **17**, 1802).**3-Phenyloxindole.**Reddish leaflets from EtOH. M.p. 183° (185-7°). Sol. EtOH,  $C_6H_6$ . Insol. acids. Sol. KOH.Aq.

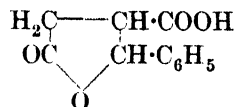
N-Acetyl: cryst. from EtOH.Aq. M.p. 103°.

Brunner, *Monatsh.*, 1897, **18**, 547.Meisenheimer, Camparter, *Ber.*, 1924, **57**, 297.**7-Phenylpalmitic Acid** $C_{22}H_{36}O_2$ 

MW, 332

B.p. 190-5°/0.12 mm.  $D_{25}^{25}$  0.9417.  $n_D^{20}$  1.4912.Et ester:  $C_{24}H_{40}O_2$ . MW, 360. B.p. 174-80°/0.13 mm.  $D_{25}^{25}$  0.9194.  $n_D^{20}$  1.4808.

p-Bromophenacyl ester: m.p. 77-8°.

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, **54**, 2515. **$\gamma$ -Phenylparaconic Acid** (3-Phenyl- $\gamma$ -butyrolactone-2-carboxylic acid) $C_{11}H_{10}O_4$ 

MW, 206

d-.

Needles +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p. anhyd. 134°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , hot  $H_2O$ , hot  $C_6H_6$ . Insol.  $CS_2$ , pet. ether.  $[\alpha]_D^{20} + 64.3^\circ$  in EtOH.

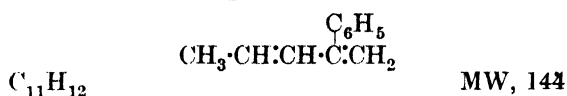
l-.

Needles +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p. anhyd. 134°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , hot  $H_2O$ , hot  $C_6H_6$ . Insol.  $CS_2$ , pet. ether.  $[\alpha]_D^{20} - 65.3^\circ$  in EtOH.

dl-.

Needles +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p. 99°, anhyd. 121°.  $k = 4.80 \times 10^{-4}$  at 20°. Heat of comb. of hydrate,  $C_r$  1195.9 Cal.Me ester:  $C_{12}H_{12}O_4$ . MW, 220. Cryst. from  $Et_2O$ . M.p. 69-70°. B.p. 211°/14 mm.Et ester:  $C_{13}H_{14}O_4$ . MW, 234. Oil with aromatic odour. B.p. 241-2°/52 mm., 224°/25 mm.Barbier, Locquin, *Bull. soc. chim.*, 1912, **13**, 233.Fittig, Jehl, *Ann.*, 1904, **330**, 345.**5-Phenyl-1 : 2-pentadiene** ( $\gamma\delta$ -Pentadienylbenzene) $C_6H_5 \cdot CH_2 \cdot CH_2 \cdot CH : C : CH_2$   
 $C_{11}H_{12}$  MW, 144B.p. 76-7°/7 mm.  $D_4^{20}$  0.9169.  $n_D^{20}$  1.5400.Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, **55**, 2816.**1-Phenyl-1 : 3-pentadiene** ( $\alpha\gamma$ -Pentadienylbenzene) $CH_2 \cdot CH : CH \cdot CH : CH \cdot C_6H_5$   
 $C_{11}H_{12}$  MW, 144Viscous oil. B.p. 240-60°, 116°/16 mm.  $D_4^{15.2}$  0.9325.  $n_D^{15.2}$  1.6000.Auwers, Eisenlohr, *J. prakt. Chem.*, 1911, **84**, 43.Pestemer, Wiligot, *Monatsh.*, 1935, **66**, 123.

## 2-Phenyl-1 : 3-pentadiene



B.p. 85–6°/15 mm. Readily polymerises.

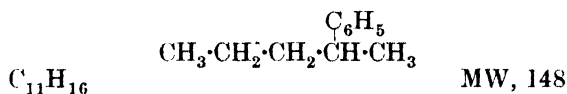
Kuhn, Hoffer, *Ber.*, 1933, **66**, 1269.

## 3-Phenylpentandione-2 : 4.

See Phenylacetylacetone.

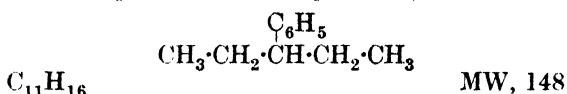
## 1-Phenylpentane.

See *n*-Amylbenzene.

2-Phenylpentane (*ω*-Methylethyltoluene)

B.p. 191–3°.  $D_4^{21}$  0.8594.  $n_D^{21}$  1.4875.

Klages, *Ber.*, 1902, **35**, 3509.

3-Phenylpentane (*Diethylphenylmethane*, *sec.*-*n*-*amylbenzene*, *ω*-*diethyltoluene*)

B.p. 180° (187°), 83–5°/22 mm., 38–41°/0.7 mm.  $D_4^{21}$  0.8755.  $n_D^{22}$  1.4868.

Levene, Marker, *J. Biol. Chem.*, 1935, **108**, 417.

## Phenylpentanol-1.

See Phenyl-*n*-amyl Alcohol.

## 1-Phenylpentanol-2.

See Propylbenzylcarbinol.

## 1-Phenylpentanol-4.

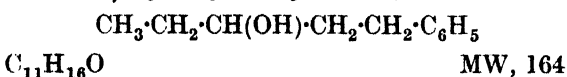
See 5-Phenyl-*sec.*-*n*-amyl alcohol.

## 2-Phenylpentanol-2.

See Methylpropylphenylcarbinol.

## 5-Phenylpentanol-2.

See 5-Phenyl-*sec.*-*n*-amyl Alcohol.

1-Phenylpentanol-3 (*Ethyl-β-phenylethylcarbinol*, *γ-hydroxy-n-amylbenzene*)

*d.*

M.p. 38°. B.p. 143°/19 mm.  $D_4^{20}$  0.9687,  $D_4^{40}$  0.8773.  $[\alpha]_{\text{D}^{40}}^{20} + 31.8^\circ$  in EtOH.

*Acid phthalate*: viscous gum.  $[\alpha]_{\text{D}^{40}}^{20} + 42.9^\circ$  in EtOH. *Strychnine salt*: m.p. 158°.  $[\alpha]_{\text{D}^{40}}^{20} - 38.7^\circ$  in  $\text{CHCl}_3$ .

*Formyl*: b.p. 135°/15 mm.  $D_4^{20}$  0.9980.  $n_{\text{D}^{40}}^{20}$  1.5003.  $[\alpha]_{\text{D}^{40}}^{20} - 13.93^\circ$ .

*Acetyl*: b.p. 147°/19 mm.  $D_4^{20}$  0.9829.  $n_{\text{D}^{40}}^{20}$  1.4920.  $[\alpha]_{\text{D}^{40}}^{20} - 11.20^\circ$ .

*l.*

M.p. 38°. B.p. 143°/19 mm.

*Acid phthalate*:  $[\alpha]_{\text{D}^{40}}^{20} - 42.9^\circ$  in EtOH. *Cinchonidine salt*: m.p. 154°.  $[\alpha]_{\text{D}^{40}}^{20} - 54.2^\circ$  in EtOH.

*Propionyl*: b.p. 150°/14 mm.  $D_4^{20}$  0.9731.  $n_{\text{D}^{40}}^{20}$  1.4878.  $[\alpha]_{\text{D}^{40}}^{20} + 22.47^\circ$ .

*dl.*

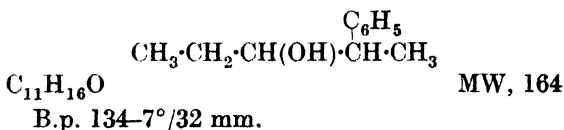
Liq. B.p. 143°/19 mm., 130°/15 mm.

*Acid phthalate*: prisms from AcOH. M.p. 74°.

*Phenylurethane*: m.p. 74°.

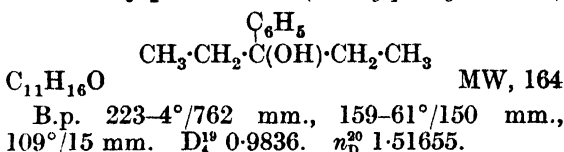
Hewitt, Kenyon, *J. Chem. Soc.*, 1925, 1094.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 151.

2-Phenylpentanol-3 (*Ethyl-α-phenylethylcarbinol*)

B.p. 134–7°/32 mm.

Tiffeneau, Lévy, *Bull. soc. chim.*, 1923, **33**, 759.

3-Phenylpentanol-3 (*Diethylphenylcarbinol*)

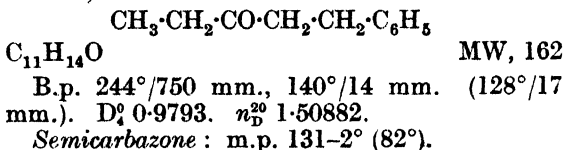
B.p. 223–4°/762 mm., 159–61°/150 mm., 109°/15 mm.  $D_4^{19}$  0.9836.  $n_D^{20}$  1.51655.

Klages, *Ber.*, 1903, **36**, 3692.

Gilman, Fothergill, Parker, *Rec. trav. chim.*, 1929, **48**, 748.

## 1-Phenylpentanone-2.

See Propyl benzyl Ketone.

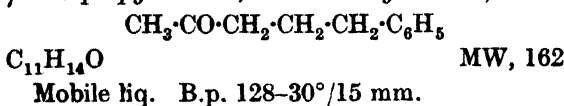
1-Phenylpentanone-3 (*γ-Keto-n-amylbenzene*)

B.p. 244°/750 mm., 140°/14 mm. (128°/17 mm.).  $D_4^{20}$  0.9793.  $n_D^{20}$  1.50882.

*Semicarbazone*: m.p. 131–2° (82°).

Hewitt, Kenyon, *J. Chem. Soc.*, 1925, 1096.

Maxim, *Ann. chim.*, 1928, **9**, 55.

1-Phenylpentanone-4 (5-Phenylpentanone-2, *γ-acetopropylbenzene*, *δ-keto-n-amylbenzene*)

Mobile liq. B.p. 128–30°/15 mm.

Semicarbazone: leaflets from MeOH. M.p. 125°.

Diels, Poetsch, *Ber.*, 1921, **54**, 1585.

Heilbron, Heslop, Irving, Wilson, *J. Chem. Soc.*, 1931, 1340.

**2-Phenylpentanone-3** ( $\omega$ -Methylbutyryl-toluene)

$\text{C}_{11}\text{H}_{14}\text{O}$   $\text{CH}_3\cdot\text{CH}_2\cdot\text{CO}\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}_3$  MW, 162

B.p. 225–8° (222–5°).  $D_4^{20}$  0.982.

Semicarbazone: m.p. 136°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

Lévy, Tabart, *Bull. soc. chim.*, 1931, **49**, 1776.

**2-Phenylpentanone-4** (4-Phenylpentanone-2,  $\beta$ -acetoisopropylbenzene)

$\text{C}_{11}\text{H}_{14}\text{O}$   $\text{CH}_3\cdot\text{CO}\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}_3$  MW, 162

B.p. 109–10°/11 mm.  $D_4^{20}$  0.9708.  $n_D^{20}$  1.51237.

Oxime: viscous liq. B.p. 160°/20 mm.

Semicarbazone: m.p. 145° (137°).

Rupe, Wild, *Ann.*, 1917, **414**, 124.

Nenitzescu, Gavât, *Ann.*, 1935, **519**, 260.

**3-Phenylpentanone-2** ( $\omega$ -Ethylacetyl-toluene)

$\text{C}_{11}\text{H}_{14}\text{O}$   $\text{CH}_3\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CO}\cdot\text{CH}_3$  MW, 162

B.p. 220–5° (215–20°).  $D_4^{20}$  0.979. Does not form bisulphite comp.

Semicarbazone: m.p. 189–90°.

Lévy, Jullien, *Bull. soc. chim.*, 1929, **45**, 941.

Tiffeneau, Lévy, Jullien, *Bull. soc. chim.*, 1931, **49**, 1788.

**1-Phenyl-1-pentene** ( $\alpha$ -Phenyl- $\alpha$ -amylene,  $\alpha$ -pentenylbenzene)

$\text{C}_{11}\text{H}_{14}$   $\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}\cdot\text{C}_6\text{H}_5$  MW, 146

B.p. 217° (202–5°), 94°/14 mm., 87.5°/9 mm.  $D_4^{20}$  0.8782.  $n_D^{20}$  1.5158.

Nitrosite: m.p. 121°.

Dibromide: m.p. 61°.

v. Braun, Köhler, *Ber.*, 1918, **51**, 79.

Prévost, Daujat, *Bull. soc. chim.*, 1930, **47**, 588.

Lévy, Dvovleitzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1765.

**2-Phenyl-1-pentene** ( $\beta$ -Phenyl- $\alpha$ -amylene)

$\text{C}_{11}\text{H}_{14}$   $\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{C}}\cdot\text{CH}_2$  MW, 146

B.p. 198–202°.  $D_4^{20}$  0.9138.

Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 357.

**3-Phenyl-1-pentene** ( $\gamma$ -Phenyl- $\alpha$ -amylene, ameylbenzene)

$\text{C}_{11}\text{H}_{14}$   $\text{CH}_3\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}\cdot\text{CH}_2$  MW, 146

B.p. 191.5° (173°), 71°/12 mm.  $D_4^{20}$  0.8458.  $n_D^{20}$  1.5030.

Dafert, *Monatsh.*, 1883, **4**, 621.

Prévost, Daujat, *Bull. soc. chim.*, 1930, **47**, 588.

**5-Phenyl-1-pentene** ( $\epsilon$ -Phenyl- $\alpha$ -amylene,  $\delta$ -pentenylbenzene)

$\text{C}_{11}\text{H}_{14}$   $\text{C}_6\text{H}_5\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}_2$  MW, 146

B.p. 203–4°, 77–8°/10 mm.  $D_4^{20}$  0.8889.  $n_D^{20}$  1.5065.

v. Braun, Deutsch, Schmatloch, *Ber.*, 1912, **45**, 1255.

**1-Phenyl-2-pentene** ( $\alpha$ -Phenyl- $\beta$ -amylene,  $\beta$ -pentenylbenzene)

$\text{C}_{11}\text{H}_{14}$   $\text{CH}_3\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{CH}\cdot\text{CH}_2\cdot\text{C}_6\text{H}_5$  MW, 146

B.p. 201°, 111°/30 mm., 97–8°/19 mm.  $D_4^{20}$  0.8884.  $n_D^{20}$  1.5089. Alc. KOH at 150°  $\rightarrow$  1-phenyl-1-pentene.

Auwers, Roth, Eisenlohr, *Ann.*, 1910, **373**, 284.

Prévost, Daujat, *Bull. soc. chim.*, 1930, **47**, 588.

**2-Phenyl-2-pentene** ( $\beta$ -Phenyl- $\beta$ -amylene)

$\text{C}_{11}\text{H}_{14}$   $\text{CH}_3\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{CH}}\cdot\text{C}\cdot\text{CH}_3$  MW, 146

Oil. B.p. 203–5°, 90°/16 mm.  $D_4^{20}$  0.8950.  $n_D^{20}$  1.5196. Acid  $\text{KMnO}_4 \rightarrow$  acetophenone.

$\text{Na} + \text{EtOH} \rightarrow$  2-phenylpentane.

Klages, *Ber.*, 1902, **35**, 3509.

Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 363.

**3-Phenyl-2-pentene** ( $\gamma$ -Phenyl- $\beta$ -amylene)

$\text{C}_{11}\text{H}_{14}$   $\text{CH}_3\cdot\text{CH}_2\cdot\overset{\text{C}_6\text{H}_5}{\text{C}}\cdot\text{CH}\cdot\text{CH}_3$  MW, 146

B.p. 197–8°/753 mm., 92°/18 mm.  $D_4^{14}$  0.9173.  $n_D^{16}$  1.5266.

*Nitroschloride*: needles. M.p. 117°.

Klages, *Ber.*, 1903, **36**, 3692.

**5-Phenyl-2-pentene** ( $\epsilon$ -Phenyl- $\beta$ -amylene,  $\gamma$ -pentenylbenzene)



$C_{11}H_{14}$  MW, 146

B.p. 203–4°.

I.G., E.P., 315,312, (*Chem. Zentr.*, 1930, I, 2161).

**Phenyl-peri Acid.**

See N-Phenyl-1-naphthylamine-8-sulphonic Acid.

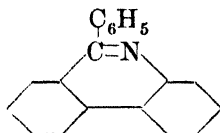
**Phenylphenacyl acetic Acid.**

See 1-Phenyl-2-benzoylpropionic Acid.

**Phenyl phenacyl Ketone.**

See Dibenzoylmethane.

**9-Phenylphenanthridine**



$C_{19}H_{13}N$  MW, 255

Plates from EtOH. M.p. 109°. B.p. above 400°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. hot H<sub>2</sub>O. Spar. sol. ligroin. Volatile in steam. Salts easily hyd. by H<sub>2</sub>O.

*B.HCl*: needles. M.p. 220°.

*B.HCl, H<sub>2</sub>O*: cryst. from dil. HCl. M.p. 95–6°.

*Nitrate*: needles. M.p. 205°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>, 2H<sub>2</sub>O*: yellow needles. Decomp. about 300° without melting.

*Picrate*: yellow needles from EtOH. M.p. 242° decomp.

Pictet, Hubert, *Ber.*, 1896, **29**, 1183, 1187.

**N-Phenyl-p-phenetidine.**

See under 4-Hydroxydiphenylamine.

**Phenylphenol.**

See Hydroxydiphenyl.

**Phenylphenylenediamine.**

See Aminodiphenylamine.

**Phenyl phenylethyl Ether.**

See under 2-Phenylethyl Alcohol.

**Phenyl phenylethyl Ketone.**

See  $\omega$ -Benzylacetophenone.

**$\alpha$ -Phenyl-phenylglycine.**

See  $\alpha$ -Anilinophenylacetic Acid.

**Phenylphosphine** (*Phosphaniline*)



$C_6H_7P$

MW, 110

B.p. 160–1°.  $D^{18}$  1.001. Oxidises very rapidly in air.

*B.HI*: needles. M.p. 138°. Decomp. by H<sub>2</sub>O into its components.

Köhler, Michaelis, *Ber.*, 1877, **10**, 808.

**Phenylphosphorous Acid**



$C_6H_7O_3P$  MW, 158

Leaflets from H<sub>2</sub>O. M.p. 158°. Sol. EtOH, Et<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>. Stable in air.

*Di-Me ester*:  $C_8H_{11}O_3P$ . MW, 186. B.p. 247°, 101–2°/15 mm.  $D_4^{20}$  1.0849.  $n_D^{20}$  1.5280.

*Et ester*:  $C_8H_{11}O_3P$ . MW, 186. Syrup. Decomp. on dist. *Anilide*: m.p. about 105°. Hygroscopic.

*Di-Et ester*:  $C_{10}H_{15}O_3P$ . MW, 214. Viscous liq. B.p. 267°.

*Phenyl ester*:  $C_{12}H_{11}O_3P$ . MW, 234. Needles from EtOH.Aq. M.p. 57°. *Anilide*: yellow cryst. M.p. 83°. B.p. 235°/25 mm.

*Diphenyl ester*:  $C_{18}H_{15}O_3P$ . MW, 310. Needles from EtOH.Aq. M.p. 63.5°.

*Dichloride*:  $C_6H_5OCl_2P$ . MW, 195. Viscous liq. B.p. 258°.  $D_4^{20}$  1.375.

*Diamide*:  $C_6H_9ON_2P$ . MW, 156. Leaflets from EtOH. M.p. 189°.

*Anhydride*: phosphinobenzene.  $C_6H_5O_2P$ . MW, 140. Cryst. powder from C<sub>6</sub>H<sub>6</sub>. M.p. 100°.

*Anilide*: m.p. 125°.

*Dianilide*: needles from EtOH. M.p. 211°.

*Di-p-toluidide*: needles from EtOH. M.p. 223°.

*Di-phenylhydrazide*: needles from EtOH. M.p. 175°.

*Aniline salt*: needles. M.p. 212°.

Arbusow, *Chem. Zentr.*, 1936, I, 543.

Michaelis, *Ann.*, 1876, **181**, 305,335; 1896, **293**, 217.

Michaelis, Köhler, *Ber.*, 1876, **9**, 521.

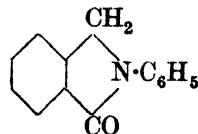
**4-Phenylphthalic Acid.**

See Diphenyl-3 : 4-dicarboxylic Acid.

**N-Phenylphthalimide.**

See Phthalanil.

**N-Phenylphthalimidine** (*Phthalidanil*)



$C_{14}H_{11}ON$  MW, 209

Leaflets from EtOH. M.p. 162–3° (160°). Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O. Very spar. sol. boiling H<sub>2</sub>O. Insol. dil. acids and alkalis.

### 3-Phenylphthalimidine

CrO<sub>3</sub> in AcOH → phthalanil. Alk. KMnO<sub>4</sub> → phthalanilic acid.

*Anil*: needles from Et<sub>2</sub>O. M.p. 142–3°. *Hydrochloride*: m.p. 237–8°. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: reddish-yellow cryst. from H<sub>2</sub>O. M.p. 212–13° decomp.

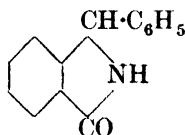
Rowe, Levin, Burns, Davies, Tepper, *J. Chem. Soc.*, 1926, 704.

Fischer, Wolter, *J. prakt. Chem.*, 1910, 80, 110.

Thiele, Schneider, *Ann.*, 1909, 369, 297.

Graebe, Pictet, *Ann.*, 1888, 247, 306.

### 3-Phenylphthalimidine



C<sub>14</sub>H<sub>11</sub>ON

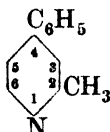
MW, 209

Cryst. from 75% AcOH. M.p. 218–20°. Sol. MeOH, EtOH. Spar. sol. Et<sub>2</sub>O. Insol. pet. ether.

*N-Acetyl*: cryst. from AcOH. M.p. 153–5°.

Rose, *J. Am. Chem. Soc.*, 1911, 33, 390.

### 4-Phenyl-α-picoline (2-Methyl-4-phenylpyridine)



C<sub>12</sub>H<sub>11</sub>N

MW, 169

Liq. resembling diphenylamine in odour. B.p. 280–90°. Volatile in steam. Turns brown in air.

B.HClO<sub>4</sub>: m.p. 142–3°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: orange-yellow needles. M.p. 211–13°.

B.HAuCl<sub>4</sub>: m.p. 161–3°.

*Picrate*: yellow needles. M.p. 210–13° (203°).

Gohdes, *J. prakt. Chem.*, 1929, 123, 184.

### 6-Phenyl-α-picoline (2-Methyl-6-phenylpyridine)

Liq. B.p. 280–1°. Volatile in steam.

B.HAuCl<sub>4</sub>: yellow needles. M.p. 150–1°. Spar. sol. cold H<sub>2</sub>O.

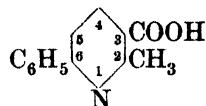
B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>.H<sub>2</sub>O: reddish needles from H<sub>2</sub>O. M.p. 200° decomp.

*Picrate*: yellow needles. M.p. 135°.

Scholtz, *Ber.*, 1895, 28, 1726.

### 438 6-Phenyl-α-picoline-3 : 4-dicarboxylic Acid

#### 6-Phenyl-α-picoline-3-carboxylic Acid (2-Methyl-6-phenylpicotinic acid)



C<sub>13</sub>H<sub>11</sub>O<sub>2</sub>N

MW, 213

Cryst. from EtOH. M.p. 196°. Sol. Me<sub>2</sub>CO, AcOEt. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Readily sol. dil. acids.

B.HCl: m.p. 288°.

*Et ester*: C<sub>15</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 241. M.p. 46–46.5°. B.p. 185°/13 mm., 160–1°/2 mm.

*Chloroplatinate*: m.p. 196°.

*Methylbetaine*: m.p. 240° decomp. B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>: prisms from EtOH. M.p. 223–4°. B.HAuCl<sub>4</sub>: yellow needles. M.p. 160–1°.

Mumm, Neumann, *Ber.*, 1926, 59, 1620.

Späth, Burger, *Monatsh.*, 1928, 49, 265.

Nienburg, *Ber.*, 1935, 68, 1475.

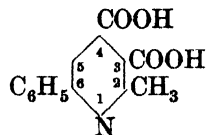
#### 6-Phenyl-α-picoline-4-carboxylic Acid (2-Methyl-6-phenylisonicotinic acid)

Cryst. from EtOH. M.p. 272°. Sol. EtOH, Me<sub>2</sub>CO, AcOEt. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Sol. dil. HCl.

*Et ester*: b.p. 194°/16 mm. Solidifies to a mass of needles.

Mumm, Neumann, *Ber.*, 1926, 59, 1621.

#### 6-Phenyl-α-picoline-3 : 4-dicarboxylic Acid (2-Methyl-6-phenylcinchomeronic acid)



C<sub>14</sub>H<sub>11</sub>O<sub>4</sub>N

MW, 257

Cryst. from EtOH. M.p. 217°. Sol. H<sub>2</sub>O, EtOH, AcOH, HCl. Insol. Et<sub>2</sub>O, AcOEt, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

*3-Et ester*: C<sub>16</sub>H<sub>15</sub>O<sub>4</sub>N. MW, 285. Needles from ligroin. M.p. 145°. Sol. Me<sub>2</sub>CO, AcOEt. Spar. sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Prac. insol. H<sub>2</sub>O.

*4-Et ester*: needles from AcOEt. M.p. 185°. Mod. sol. Et<sub>2</sub>O, AcOEt, C<sub>6</sub>H<sub>6</sub>, ligroin. Spar. sol. H<sub>2</sub>O.

*Di-Et ester*: C<sub>18</sub>H<sub>19</sub>O<sub>4</sub>N. MW, 313. Needles from AcOEt. M.p. 73°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. ligroin. *Picrate*: m.p. 83°.

*4-Amide*: C<sub>14</sub>H<sub>13</sub>O<sub>3</sub>N<sub>2</sub>. MW, 256. Cryst. from EtOH. M.p. 199°. Spar. sol. org. solvents.

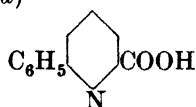
**Anhydride:**  $C_{14}H_9O_3N$ . MW, 239. Needles from  $C_6H_6$ . M.p.  $196^\circ$ . Sol.  $Et_2O$ ,  $AcOEt$ ,  $C_6H_6$ , ligroin.

**Imide:**  $C_{14}H_{10}O_2N_2$ . MW, 238. Needles from  $Me_2CO$ . M.p.  $249^\circ$ .

Mumm, Böhme, *Ber.*, 1921, **54**, 731.

Mumm, Neumann, *Ber.*, 1926, **59**, 1616.

**6-Phenylpicolinic Acid** (6-Phenylpyridine-2-carboxylic acid)

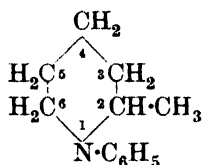


$C_{12}H_9O_2N$  MW, 199

Needles from  $H_2O$ . M.p.  $109^\circ$ . Very sol.  $EtOH$ . Heat at  $190-200^\circ \rightarrow$  2-phenylpyridine.  $FeSO_4$  in  $H_2O \rightarrow$  red col.

Scholtz, *Ber.*, 1895, **28**, 1728.

**N-Phenyl- $\alpha$ -pipecoline** (2-Methyl-N-phenylpiperidine)



$C_{12}H_{17}N$  MW, 175

B.p.  $256.5-257^\circ/710$  mm.,  $143^\circ/20$  mm. Sol.  $EtOH$ ,  $Et_2O$ . Very spar. sol.  $H_2O$ .

$B_2H_2PtCl_6$ : orange-yellow needles. Decomp. at  $212^\circ$ .

**Picrate**: yellow prisms from  $EtOH$ . M.p.  $167-8^\circ$  ( $162^\circ$ ).

**Methiodide**: cryst. powder from  $EtOH-Et_2O$ . M.p.  $145^\circ$ .

v. Braun, Sobiecki, *Ber.*, 1911, **44**, 1045.

Lipp, *Ann.*, 1896, **289**, 245.

**6-Phenyl- $\alpha$ -pipecoline** (2-Methyl-6-phenylpiperidine).

B.p.  $112-14^\circ/12$  mm.  $D_4^{25}$  0.9096.  $n_D^{25}$  1.4882.

Two inactive stereoisomeric forms are known:  $\alpha$ -.

$B, HCl$ : m.p.  $191-2^\circ$ . Sol.  $Me_2CO$ .

$B_2H_2PtCl_6$ : m.p.  $236^\circ$ .

$\beta$ -.

B.p.  $117.5-118^\circ/11$  mm.

$B, HCl$ : cryst. from  $Me_2CO$ . M.p.  $225^\circ$ .

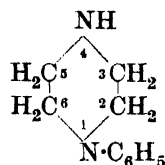
$B_2H_2PtCl_6$ : m.p.  $257^\circ$ .

Adkins, Kuick, Farlow, Wojcik, *J. Am. Chem. Soc.*, 1934, **56**, 2425.

Singer, McElvain, *J. Am. Chem. Soc.*, 1935, **57**, 1137.

Meisenheimer, Stratman, Theilacker, *Ber.*, 1932, **65**, 423.

## N-Phenylpiperazine



$C_{10}H_{14}N_2$  MW, 162

Oil. B.p.  $156-7^\circ/10$  mm.  $D_4^{25}$  1.0725.  $n_D$  1.59053.

$B, HCl$ : m.p.  $247^\circ$  decomp.

$B, HBr$ : m.p.  $250-2^\circ$ .

4-Acetyl: m.p.  $96^\circ$ .  $B, HCl$ : m.p.  $213-14^\circ$ .

4-Benzoyl: m.p.  $96-7^\circ$ .  $B, HCl$ : m.p.  $244^\circ$ .

4-p-Tolylsulphonyl: m.p.  $199-200^\circ$ .

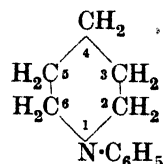
4-Benzyl: m.p.  $59^\circ$ .  $B, HCl$ : m.p.  $228^\circ$ .

Prelog, Blazek, *Chem. Abstracts*, 1935, **29**, 2959.

Prelog, Driza, *Chem. Abstracts*, 1934, **28**, 1347.

Pollard, MacDowell, *J. Am. Chem. Soc.*, 1934, **56**, 2199.

**N-Phenylpiperidine** (1-Phenylpiperidine, pentamethylene-aniline)



$C_{11}H_{15}N$  MW, 161

B.p.  $245-50^\circ$  ( $257-8^\circ/752$  mm.). Sol.  $EtOH$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

$B, HBr$ : leaflets from  $EtOH-Et_2O$ . M.p.  $235^\circ$ .

$B_2H_2PtCl_6, 2H_2O$ : leaflets or needles. Decomp. at  $190^\circ$ .

**Picrate**: yellow cryst. from  $EtOH$ . M.p.  $148^\circ$ .

**Methobromide**: cryst. from  $EtOH-Et_2O$ . M.p.  $170^\circ \rightarrow$  N-phenylpiperidine + methyl bromide. Somewhat hygroscopic.

**Methiodide**: cryst. from  $EtOH-Et_2O$ . M.p.  $146^\circ$ . Sol.  $H_2O$ ,  $EtOH$ . Gradually turns yellow in air.

Le Fèvre, *J. Chem. Soc.*, 1932, 1378.

Paul, *Bull. soc. chim.*, 1933, **53**, 1489.

v. Braun, *Ber.*, 1907, **40**, 3920.

## 2-Phenylpiperidine.

Cryst. +  $1H_2O$ . M.p.  $60-1^\circ$ . The anhyd. base is an oil, b.p.  $255-255.5^\circ/767$  mm.,  $110-12^\circ/9$  mm. Volatile in steam.

$B, HCl$ : needles from  $EtOH-Et_2O$ . M.p.  $196-7^\circ$ .

$B, H AuCl_4$ : yellow cryst. M.p.  $159-60^\circ$ .



$B_2H_2PtCl_6$ : yellow plates. Decomp. about 197°.

Picrate: cryst. from  $H_2O$ . M.p. 115–17°.

N-Benzoyl:  $BHCl$ , m.p. 186–7°.

Adkins, Kuick, Farlow, Wojcik, *J. Am.*

*Chem. Soc.*, 1934, 56, 2427.

Gabriel, *Ber.*, 1908, 41, 2013.

### 3-Phenylpiperidine.

B.p. 255–6°/740 mm.  $D_{25}^{25}$  1.0040.  $n_D^{25}$  1.5473.

$BHCl$ : m.p. 146–7°.

N-Benzoyl:  $BHCl$ , m.p. 180–1°.

Walters, McElvain, *J. Am. Chem. Soc.*,

1933, 55, 4625.

### 4-Phenylpiperidine.

M.p. 57.5–58° (50°). B.p. 255–7°/727 mm.

Insol.  $H_2O$ . Absorbs  $CO_2$  from the air.

$BHNO_3$ : plates from  $H_2O$ . M.p. 139°.

Chloroplatinate: orange leaflets. M.p. 204–7°.

N-Benzoyl: hydrochloride, m.p. 174–5°.

Forsyth, Pyman, *J. Chem. Soc.*, 1930, 401.

Adkins, Kuick, Farlow, Wojcik, *J. Am.*

*Chem. Soc.*, 1934, 56, 2427.

Bally, *Ber.*, 1887, 20, 2590.

### N-Phenylpiperidine-4-carboxylic Acid.

See N-Phenylhexahydroisonicotinic Acid.

### Phenylpiperidylmethane.

See Benzylpiperidine.

### Phenylpropandiol.

See Phenylpropylene Glycol and Phenyltrimethylene Glycol.

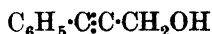
### Phenylpropane.

See Cumene and Propylbenzene.

### Phenylpropanolone.

See 3-Phenylhydroxyacetone.

**Phenylpropargyl Alcohol** (*Phenylacetylenylcarbinol, phenylpropionic alcohol, hydroxymethylphenylacetylene*)



$C_9H_8O$

MW, 132

B.p. 137–8°/15 mm. (140°/12 mm.).  $D_{15}^{15}$  1.07.  $n_D^{15}$  1.5873. Heat of comb.  $C_9$  1138.1 Cal. Reduces  $NH_3 \cdot AgNO_3$ .

Me ether:  $C_{10}H_{10}O$ . MW, 146. B.p. 226°, 115°/16 mm.  $D_4^{20}$  1.0016.  $n_D^{20}$  1.5502.

Propyl ether:  $C_{12}H_{14}O$ . MW, 174. B.p. 137°/17 mm.  $D_4^{20}$  0.9668.  $n_D^{20}$  1.5326.

Acetyl: b.p. 145–7°/16 mm.

Guest, *J. Am. Chem. Soc.*, 1925, 47, 862.

Bert, *Compt. rend.*, 1930, 191, 493.

Lai, *Bull. soc. chim.*, 1933, 53, 682.

Moureu, André, *Ann. chim.*, 1914, 1, 119.

### Phenylpropargyl Aldehyde.

See Phenylpropionic Aldehyde.

### Phenylpropionic Acid



$C_9H_8O_2$

MW, 146

Needles from  $H_2O$  or  $CS_2$ . M.p. 137°. Melts under  $H_2O$  at approx. 80°. Very sol. EtOH, Et<sub>2</sub>O.  $k = 5.9 \times 10^{-3}$  at 25°. Heat of comb.  $C_9$  1022.0 Cal.  $Zn + AcOH \rightarrow$  cinnamic acid.  $NaHg \rightarrow$  hydrocinnamic acid. Dissolving in conc.  $H_2SO_4 \rightarrow$  benzoylacetic acid. Hot baryta  $\rightarrow$  phenylacetylene.

Me ester:  $C_{10}H_8O_2$ . MW, 160. Cryst. M.p. 26°. B.p. 158–9°/48 mm., 132–3°/16 mm.  $D_4^{25}$  1.0830.  $n_D^{25}$  1.5618. Heat of comb.  $C_9$  7478 cal./gm.

Et ester:  $C_{11}H_{10}O_2$ . MW, 174. Oil. B.p. 260–70° (rapid dist.), 152–3°/21 mm., 144°/13 mm.  $D_4^{25}$  1.0550.  $n_D^{25}$  1.5502. Heat of comb.  $C_9$  7693 cal./gm.

d-Amyl ester:  $C_{14}H_{16}O_2$ . MW, 216. B.p. 210°/55 mm.  $D_4^{20}$  1.0035.  $[\alpha]_D^{20} + 5.58^\circ$ .

l-Menthyl ester: needles. M.p. 63–4°. B.p. 235–8°/30 mm.  $D_4^{25}$  1.0595.  $n_D^{25}$  1.5239.  $[\alpha]_D^{25} - 58.65^\circ$  in  $CHCl_3$ ,  $[\alpha]_D^{20} - 72.6^\circ$  in  $C_6H_6$ .

d-Bornyl ester: yellowish cryst. M.p. 45°. B.p. 228–30°/21 mm.  $D_4^{25}$  1.0884.  $n_D^{25}$  1.55.  $[\alpha]_D^{25} + 31.05^\circ$  in  $CHCl_3$ .

p-Nitrobenzyl ester: m.p. 83°.

Chloride:  $C_9H_7OCl$ . MW, 164.5. B.p. 115–16°/17 mm. (119°/12 mm.).

Amide:  $C_9H_7ON$ . MW, 145. Needles from  $H_2O$ . M.p. 108–9° (99–100°). Sol. MeOH, EtOH,  $CHCl_3$ . Spar. sol. Et<sub>2</sub>O, cold.  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  benzoylacetylacetamide. Hydrazine hydrate in EtOH  $\rightarrow$  3-phenylpyrazolone-5.

Nitrile:  $C_9H_5N$ . MW, 127. M.p. 41°. B.p. 228–9°, 105–6°/13 mm.  $D_4^{21.5}$  1.0046.  $n_D^{21.5}$  1.58535.

Hydrazide: cryst. from EtOH. M.p. 114°  $\rightarrow$  3-phenylpyrazolone-5.  $BHCl$ : hygroscopic cryst. M.p. 138–9°. Sol. EtOH, Et<sub>2</sub>O. Picrate: yellow plates +  $1H_2O$ . M.p. 105–10°.

Azide: needles from Et<sub>2</sub>O. M.p. 55°. Insol.  $H_2O$ .

Anilide: needles from EtOH. M.p. 126°.

p-Toluidide: plates from EtOH. M.p. 142°.

Abbott, *Organic Syntheses*, 1932, XII, 60.

Curtius, Kennigott, *J. prakt. Chem.*, 1926, 112, 314.

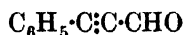
Bogert, Marcus, *J. Am. Chem. Soc.*, 1919, 41, 88 (Note 1).

Michael, *Ber.*, 1901, 34, 3648.

Moureu et al., *Ann. chim.*, 1914, 2, 276.

**Phenylpropionic Alcohol.**

See Phenylpropargyl Alcohol.

**Phenylpropionic Aldehyde** (*Phenylpropargyl aldehyde*) $\text{C}_9\text{H}_8\text{O}$ 

MW, 130

Oil with odour resembling cinnamaldehyde. B.p. 127–8°/28 mm., 118°/17 mm., 112–13°/15 mm., 104–5°/11 mm.  $D_4^{20}$  1.0680.  $n_D^{20}$  1.60785. Decomp. on dist. at atmospheric press. Heat of comb.  $\text{C}_9$  1081.7 Cal. Aq. alkalis  $\rightarrow$  phenylacetylene +  $\text{H}\cdot\text{COOH}$ .

*Oxime*: needles from ligroin. M.p. 108°. Acetic anhydride  $\rightarrow$  phenylpropionic nitrile.

*Di-Et acetal*: b.p. 144–5°/14 mm.  $D^{13}$  0.9940.  $n_D^{13}$  1.52216.

Kalf, *Rec. trav. chim.*, 1927, **46**, 594.Auwers, Seyfried, *Ann.*, 1930, **484**, 224.Moureau, Delange, *Bull. soc. chim.*, 1904, **31**, 1329.**2-Phenylpropionaldehyde.**

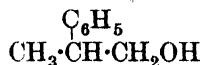
See Hydrocinnamaldehyde.

**1-Phenylpropionic Acid.**

See Hydratropic Acid.

**2-Phenylpropionic Acid.**

See Hydrocinnamic Acid.

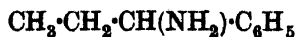
 **$\beta$ -Phenylpropiofenone.**See  $\alpha$ -Methyldeoxybenzoin.**1-Phenylpropyl Alcohol.**Ethylphenylcarbinol, *q.v.***2-Phenylpropyl Alcohol** ( $\beta$ -Hydroxyisopropylbenzene,  $\beta$ -hydroxycumene, hydratropic alcohol) $\text{C}_9\text{H}_{12}\text{O}$ 

MW, 136

dl.

B.p. 114°/14 mm.  $D^{20}$  1.017.*Acid phthalate*: prisms from  $\text{CS}_2$ . M.p. 79°.*Acetyl*: b.p. 125°/14 mm., 103–5°/10 mm.*Benzoyl*: b.p. 198–200°/20 mm.*p*-Nitrobenzoyl: plates. M.p. 65°.*Phenylurethane*: needles. M.p. 156°.Ramart, Amagat, *Ann. chim.*, 1927, **8**, 263.Cohen, Marshall, Woodman, *J. Chem. Soc.*, 1915, **107**, 897.**3-Phenylpropyl Alcohol.**

See Hydrocinnamyl Alcohol.

**1-Phenylpropylamine** ( $\alpha$ -Aminopropylbenzene) $\text{C}_9\text{H}_{13}\text{N}$ 

MW, 135

Oil. B.p. 204–6°/748 mm., 100–5°/35 mm., 99–100°/16 mm.  $D_4^{20}$  0.9560,  $D_4^{25}$  0.9347.  $n_D^{25}$  1.51726. Spar. sol.  $\text{H}_2\text{O}$ . Absorbs  $\text{CO}_2$ .

*B, HCl*: needles from EtOH. M.p. 189.5° (194°).

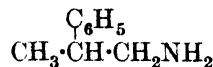
*N-Benzoyl*: needles from EtOH. M.p. 115–16°.

*N-Benzenesulphonyl*: needles from EtOH. M.p. 81°.

*N-Me*:  $\alpha$ -methylaminopropylbenzene.  $\text{C}_{10}\text{H}_{15}\text{N}$ . MW, 149. B.p. 96°/20 mm. *B, HCl*: needles or prisms. M.p. 153°.

*N-Et*:  $\alpha$ -ethylaminopropylbenzene.  $\text{C}_{11}\text{H}_{17}\text{N}$ . MW, 163. B.p. 207–8°/729 mm., 99°/20 mm. *B, HCl*: needles or prisms. M.p. 180°.

*N-Phenyl*:  $\alpha$ -phenylpropylaniline,  $\alpha$ -anilino-propylbenzene.  $\text{C}_{15}\text{H}_{17}\text{N}$ . MW, 211. Viscous oil. B.p. 192°/20 mm. *B, HCl*: needles from EtOH– $\text{Et}_2\text{O}$ . M.p. 187°. *B, HNO\_3*: needles or prisms. M.p. 174°. Part. hyd. by  $\text{H}_2\text{O}$ .

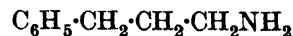
Hartung, Munch, *J. Am. Chem. Soc.*, 1931, **53**, 1878.Konowalow, *Chem. Zentr.*, 1894, **I**, 465.Busch, Leefhelm, *J. prakt. Chem.*, 1908, **77**, 8.**2-Phenylpropylamine** ( $\beta$ -Aminoisopropylbenzene,  $\beta$ -aminocumene) $\text{C}_9\text{H}_{13}\text{N}$ 

MW, 135

Oil with fishy odour. B.p. 210°, 104°/21 mm. Misc. with EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Very spar. sol.  $\text{H}_2\text{O}$ . Absorbs  $\text{H}_2\text{O}$  and  $\text{CO}_2$  from the air.

*B, HCl*: m.p. 123–4°.*B, H\_2PtCl\_6*: m.p. 229° decomp.*B, H\_2AuCl\_4*: m.p. 124°.*N-Benzoyl*: cryst. from EtOH. M.p. 85°.*Picrate*: m.p. 182°.

See first reference above and also

v. Braun, Grabowski, Kirschbaum, *Ber.*, 1913, **46**, 1281.Freund, König, *Ber.*, 1893, **26**, 2875.**3-Phenylpropylamine** ( $\gamma$ -Aminopropylbenzene) $\text{C}_9\text{H}_{13}\text{N}$ 

MW, 135

B.p. 221.5°/755 mm., 75–80°/1 mm.  $D_4^{25}$  0.9760. Misc. with EtOH,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$  with strongly alk. reaction. Absorbs  $\text{CO}_2$ .

*B, HCl*: leaflets from EtOH– $\text{Et}_2\text{O}$ . M.p. 218°.*B, H\_2PtCl\_6*: yellow leaflets from  $\text{H}_2\text{O}$ . Decomp. at 233°.*Neutral oxalate*: needles from  $\text{H}_2\text{O}$ . M.p. 190° decomp.

*Acid oxalate*: prisms from EtOH. M.p. 156°. Very sol. hot H<sub>2</sub>O.

*Picrate*: lemon-yellow needles from H<sub>2</sub>O. M.p. 152-3°.

*N-Me*:  $\gamma$ -methylaminopropylbenzene. C<sub>10</sub>H<sub>15</sub>N. MW, 149. Odourless oil. B.p. 116°/20 mm. (133-5°/18 mm.). B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: red leaflets from H<sub>2</sub>O. M.p. 188°. *Picrate*: needles from EtOH. M.p. 93-4°.

*N-Di-Me*:  $\gamma$ -dimethylaminopropylbenzene. C<sub>11</sub>H<sub>17</sub>N. MW, 163. B.p. 222-4°, 117-18°/26 mm., 99°/14 mm. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: m.p. 146°. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: orange cryst. M.p. 152°. *Methobromide*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 143°. *Methiodide*: needles from EtOH. M.p. 179°.

*N-Et*:  $\gamma$ -ethylaminopropylbenzene. C<sub>11</sub>H<sub>17</sub>N. MW, 163. B.p. 124-6°/25 mm., 118°/16 mm. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: needles. M.p. 134-5°.

*N-Di-Et*:  $\gamma$ -diethylaminopropylbenzene. C<sub>13</sub>H<sub>21</sub>N. MW, 191. B.p. 137-9°/22 mm.

*N-Propyl*:  $\gamma$ -propylaminopropylbenzene. C<sub>12</sub>H<sub>19</sub>N. MW, 177. B.p. 134°/17 mm. *Picrate*: orange cryst. from EtOH-Et<sub>2</sub>O. M.p. 97°.

*N-Dipropyl*:  $\gamma$ -dipropylaminopropylbenzene. C<sub>16</sub>H<sub>25</sub>N. MW, 219. B.p. 158-60°/17 mm. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: red cryst. M.p. 91-3°.

*N-Benzoyl*: cryst. from H<sub>2</sub>O. M.p. 57-8°.

Hartung, Munch, *J. Am. Chem. Soc.*, 1931, **53**, 1878.

Goodyear, F.P., 751,712, (*Chem. Zentr.*, 1934, I, 126).

v. Braun, Aust, *Ber.*, 1916, **49**, 504.

Emde, *Ann.*, 1912, **391**, 93.

v. Braun, *Ber.*, 1910, **43**, 3218.

Gabriel, Eschenbach, *Ber.*, 1897, **30**, 1128.

Michaelis, Jacobi, *Ber.*, 1893, **26**, 2160.

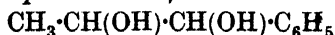
### $\alpha$ -Phenylpropylaniline.

See under 1-Phenylpropylamine.

### Phenylpropylene.

See Allylbenzene and  $\alpha$ - and  $\beta$ -Methylstyrenes.

**1-Phenylpropylene Glycol** ( $\alpha\beta$ -Dihydroxypropylbenzene,  $\alpha$ -methyl- $\alpha'$ -phenylethylene glycol, 1-phenylpropandiol-1:2)



C<sub>9</sub>H<sub>12</sub>O<sub>2</sub> MW, 152

Exists in two forms:

(i) Plates from Et<sub>2</sub>O-pet. ether. M.p. 56-7° 52-3°. Very sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. Ox.  $\rightarrow$  benzaldehyde and acetaldehyde.

*Dibenzoyl*: needles. M.p. 101°.

(ii) Plates from Et<sub>2</sub>O. M.p. 92-3°. 20% H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  phenylacetone. Ox.  $\rightarrow$  benzaldehyde and acetaldehyde.

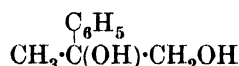
*Dibenzoyl*: m.p. 76-7°.

Lévy, Droleitzka-Gombinska, *Bull. soc. chim.*, 1931, **49**, 1765.

Zincke, Zahn, *Ber.*, 1910, **43**, 851.

Tiffeneau, *Compt. rend.*, 1906, **142**, 1538.

**2-Phenylpropylene Glycol** ( $\alpha\beta$ -Dihydroxyisopropylbenzene,  $\alpha$ -methyl- $\alpha$ -phenylethylene glycol, 2-phenylpropandiol-1:2)



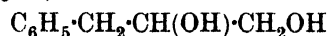
C<sub>9</sub>H<sub>12</sub>O<sub>2</sub> MW, 152

Needles from pet. ether. or ligroin-Et<sub>2</sub>O. M.p. 44-5° (40-1°). B.p. 158-60°/25 mm., 153°/16 mm. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. ligroin.

Danilow, Venus-Danilowa, *Ber.*, 1927, **60**, 1063.

Stoermer, *Ber.*, 1906, **39**, 2297.

**3-Phenylpropylene Glycol** ( $\beta\gamma$ -Dihydroxypropylbenzene, 3-phenylpropandiol-1:2, benzylethylene glycol)



C<sub>9</sub>H<sub>12</sub>O<sub>2</sub> MW, 152

B.p. 163-5°/15 mm.

*Diacyl*: b.p. 282-6°, 163-6°/13 mm. D° 1.128.

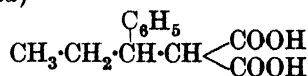
*Dibenzoyl*: m.p. 74-5°.

1-Phenyl ether: C<sub>15</sub>H<sub>16</sub>O<sub>2</sub>. MW, 228. Plates from C<sub>6</sub>H<sub>6</sub>. M.p. 91-2°. Insol. H<sub>2</sub>O.

Hershberg, *Helv. Chim. Acta*, 1934, **17**, 351

Fourneau, *Chem. Zentr.*, 1910, I, 1134.

$\alpha$ -Phenylpropyl-malonic Acid (2-Phenylbutane-1:1-dicarboxylic acid, ethylphenylis succinic acid)



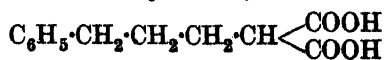
C<sub>12</sub>H<sub>14</sub>O<sub>4</sub> MW, 222

Plates + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 74°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. At 150°  $\rightarrow$  2-phenylvaleric acid.

*Di-Et ester*: C<sub>16</sub>H<sub>22</sub>O<sub>4</sub>. MW, 278. B.p. 187-8°/22 mm.

Reynolds, *Am. Chem. J.*, 1910, **44**, 315.

$\gamma$ -Phenylpropyl-malonic Acid (4-Phenylbutane-1:1-dicarboxylic acid)



C<sub>12</sub>H<sub>14</sub>O<sub>4</sub> MW, 222

Cryst. from  $C_6H_6$ -ligroin. M.p.  $98^\circ$  ( $94^\circ$ ). Sol. most org. solvents except ligroin. Heated above m.p. or better at  $200^\circ$  under reduced press.  $\rightarrow$  4-phenylvaleric acid.

*Di-Me ester*:  $C_{14}H_{18}O_4$ . MW, 250. B.p.  $183-4^\circ/10$  mm.

*Di-Et ester*: b.p.  $189-94^\circ/13$  mm.

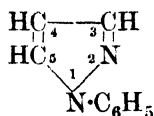
*Et ester-nitrile*:  $C_{14}H_{17}O_2N$ . MW, 231. Oil. B.p.  $192-3^\circ/11$  mm.

v. Braun, Kruber, *Ber.*, 1912, 45, 386.

Przewalski, *Chem. Zentr.*, 1923, III, 665.

Borsche, *Ber.*, 1912, 45, 622.

### 1-Phenylpyrazole



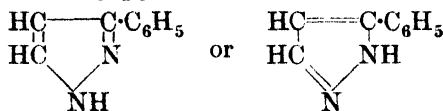
$C_9H_8N_2$  MW, 144

Needles. M.p.  $11-11.5^\circ$ . B.p.  $246-7^\circ$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol. warm H<sub>2</sub>O.

*Chloroaurate*: yellow needles from EtOH-HCl. M.p.  $181^\circ$ .

Alvisi, *Gazz. chim. ital.*, 1892, 22, i, 161.

### 3(5)-Phenylpyrazole



$C_9H_8N_2$  MW, 144

Needles from H<sub>2</sub>O. M.p.  $79^\circ$ . B.p.  $313-14^\circ$ ,  $177-8^\circ/11$  mm. Sol. EtOH, Et<sub>2</sub>O.  $D_4^{20}$  1.0818.  $n_{D,20}^{100}$  1.58890.

*B,HCl*: cryst. powder. M.p.  $144-5^\circ$ .

*B,HNO<sub>3</sub>*: needles from Et<sub>2</sub>O. M.p.  $126^\circ$ , decomp. Sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O<sup>o</sup> Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>.

*N-Acetyl*: needles from ligroin. M.p.  $64-5^\circ$ . B.p.  $157-8^\circ/10$  mm. Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, ligroin.  $D_4^{100}$  1.0778.  $n_{D,20}^{100}$  1.56064.

*N-o-Nitrobenzoyl of 3-phenylpyrazole*: cryst. from MeOH. M.p.  $107.5-108.5^\circ$ . Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. MeOH.

*N-o-Nitrobenzoyl of 5-phenylpyrazole*: needles from MeOH. M.p.  $151-2^\circ$ . Sol. Me<sub>2</sub>CO. Spar. sol. MeOH, C<sub>6</sub>H<sub>6</sub>.

Auwers, Dietrich, *J. prakt. Chem.*, 1934, 139, 89.

Auwers, Cauver, *J. prakt. Chem.*, 1930, 126, 194.

Auwers, Schmidt, *Ber.*, 1925, 58, 538.

Buchner, Hachumian, *Ber.*, 1902, 35, 38.

For non-equivalence of positions 3 and 5 see Auwers, *Ann.*, 1933, 508, 57.

### 4-Phenylpyrazole.

Plates from EtOH. M.p.  $230^\circ$ . Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O.

*B,HCl*: needles. M.p.  $215-18^\circ$ .

*N-Acetyl*: yellow needles from EtOH. M.p.  $81.5-82.5^\circ$ . B.p.  $159-61^\circ$ . Sol. Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin.

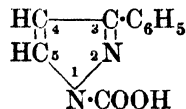
*Picrate*: yellow cryst. from EtOH. M.p.  $155^\circ$ .

Auwers, Cauver, *J. prakt. Chem.*, 1930, 126, 194.

Rupe, Huber, *Helv. Chim. Acta*, 1927, 10, 848.

Buchner, Perkel, *Ber.*, 1903, 36, 3777.

### 3-Phenylpyrazole-1-carboxylic Acid



$C_{10}H_8O_2N_2$  MW, 188

*Et ester*:  $C_{12}H_{12}O_2N_2$ . MW, 216. B.p.  $193^\circ/13$  mm.  $D_4^{10}$  1.1694.  $n_{D,20}^{10}$  1.57904.

*Amide*:  $C_{10}H_9ON_3$ . MW, 187. Needles from Me<sub>2</sub>CO. M.p.  $142-3^\circ$ . Sol. ord. org. solvents. *Picrate*: yellow needles from Et<sub>2</sub>O. M.p.  $165-6^\circ$ .

Auwers, Dietrich, *J. prakt. Chem.*, 1934, 139, 89.

Auwers, Cauver, *J. prakt. Chem.*, 1930, 126, 197.

Auwers, Ottens, *Ber.*, 1925, 58, 2076.

### 4-Phenylpyrazole-1-carboxylic Acid.

*Et ester*: plates from ligroin. M.p.  $78-78.5^\circ$ . B.p.  $192^\circ/9$  mm. Very sol. ord. org. solvents.  $D_4^{20}$  1.1055.  $n_{D,20}^{99.5}$  1.54637.

Auwers, Cauver, *J. prakt. Chem.*, 1930, 126, 193.

### 5-Phenylpyrazole-1-carboxylic Acid.

*Me ester*:  $C_{11}H_{10}O_2N_2$ . MW, 202. Needles from EtOH. M.p.  $152^\circ$ . Sol. ord. org. solvents.

*Et ester*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p.  $58-9^\circ$ . Very sol. MeOH, EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>.  $D_4^{78.4}$  1.1165.  $n_{D,20}^{78.4}$  1.53860.

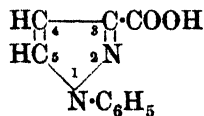
*Amide*: plates from C<sub>6</sub>H<sub>6</sub>. M.p.  $133-4^\circ$ . Very sol. AcOH. Mod. sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O.

Auwers, Breyhan, *J. prakt. Chem.*, 1935, 143, 267.

Auwers, Dietrich, *J. prakt. Chem.*, 1934, 139, 89.

Auwers, Ottens, *Ber.*, 1925, 58, 2077.

### 1-Phenylpyrazole-3-carboxylic Acid



$C_{10}H_8O_2N_2$  MW, 188

**4-Phenylpyrazole-3-carboxylic Acid**

444

Needles from  $\text{H}_2\text{O}$ . M.p.  $146^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_{11}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 202. Needles. M.p.  $77^\circ$ .

Claisen, Roosen, *Ann.*, 1894, **278**, 294.

**4-Phenylpyrazole-3-carboxylic Acid.**

Plates. M.p.  $252-3^\circ$  with decomp. to 4-phenylpyrazole. Spar. sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Me}_2\text{CO}$ . Insol.  $\text{CHCl}_3$ .

*Me ester*: prisms from MeOH. M.p.  $188-90^\circ$ . *N-Acetyl*: needles from EtOH. M.p.  $129.5-130.5^\circ$ . Sol.  $\text{Et}_2\text{O}$ . Mod. sol. EtOH. Spar. sol. ligroin.

*Et ester*:  $\text{C}_{12}\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 216. Plates from EtOH. M.p.  $164-5^\circ$ . Sol. MeOH. Spar. sol.  $\text{Et}_2\text{O}$ .

*Nitrile*: needles from  $\text{C}_6\text{H}_6$ . M.p.  $149.5-150^\circ$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin.

Auwers, Ungemach, *Ber.*, 1933, **66**, 1204, 1207.

Auwers, Cauver, *Ann.*, 1929, **470**, 302.

Kohler, Steele, *J. Am. Chem. Soc.*, 1919, **41**, 1104.

**5(3) - Phenylpyrazole - 3(5) - carboxylic Acid.**

Needles from  $\text{H}_2\text{O}$  or EtOH. M.p.  $234^\circ$  decomp. Sol. MeOH, EtOH. Insol.  $\text{C}_6\text{H}_6$ .

*Me ester*:  $\text{C}_{11}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 202. Prisms from MeOH or  $\text{Et}_2\text{O}$ . M.p.  $182^\circ$ .

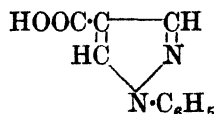
*Et ester*:  $\text{C}_{12}\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 216. Needles from MeOH or  $\text{Et}_2\text{O}$ . M.p.  $140^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Very sol.  $\text{CHCl}_3$ . *N-Acetyl*: needles from  $\text{C}_6\text{H}_6$ . M.p.  $109-10^\circ$ .

*Hydrazide*: m.p.  $205^\circ$ .

Auwers, Dietrich, *J. prakt. Chem.*, 1934, **139**, 93.

Auwers, Mausolf, *Ber.*, 1927, **60**, 1732.

Bülow, *Ber.*, 1904, **37**, 2201.

**1-Phenylpyrazole-4-carboxylic Acid**

$\text{C}_{10}\text{H}_8\text{O}_2\text{N}_2$

MW, 188

Needles from  $\text{H}_2\text{O}$ . M.p.  $219-20^\circ$ . Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Sublimes in needles.

*Me ester*: needles from MeOH. M.p.  $128-9^\circ$ .

*Et ester*: prisms from EtOH. M.p.  $96-7^\circ$ .

Wislicenus, Bindemann, *Ann.*, 1901, **316**, 36.

**3-Phenylpyrazole-1 : 4-dicarboxylic Acid****5-Phenylpyrazole-4-carboxylic Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $260^\circ$ . Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ .

*Me ester*: needles from ligroin. M.p.  $111.5-112.5^\circ$ .

*Et ester*: needles from EtOH.Aq. M.p.  $85-6^\circ$ .

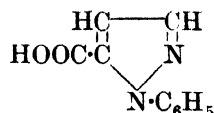
*Anilide*: cryst. + 1EtOH from EtOH. M.p.  $182^\circ$ .

*o-Anisidide*: needles from EtOH.Aq. M.p.  $127^\circ$ . Sol. most org. solvents.

*p-Anisidide*: needles from EtOH.Aq. M.p.  $161^\circ$ .

Auwers, Ungemach, *Ber.*, 1933, **66**, 1207.

Dains, Long, *J. Am. Chem. Soc.*, 1921, **43**, 1201.

**1-Phenylpyrazole-5-carboxylic Acid**

$\text{C}_{11}\text{H}_8\text{O}_4\text{N}_2$

MW, 232

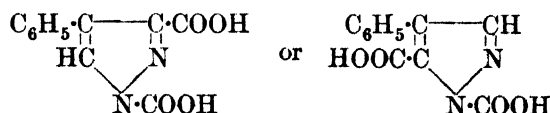
Needles from  $\text{H}_2\text{O}$ . M.p.  $183^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_{11}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 202. Needles. M.p.  $67^\circ$ .

*Chloride*: needles from ligroin. M.p.  $53^\circ$ . B.p.  $152/15$  mm.

Rojahn, Seitz, *Ann.*, 1924, **437**, 303.

Claisen, Roosen, *Ann.*, 1894, **278**, 292.

**4 - Phenylpyrazole - 1 : 3(5) - dicarboxylic Acid**

$\text{C}_{11}\text{H}_8\text{O}_4\text{N}_2$

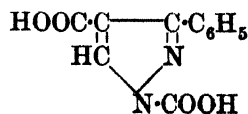
MW, 232

3(5)-*Et ester*:  $\text{C}_{13}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 260.

*Anilide*: needles from ligroin. M.p.  $105-6^\circ$ . Sol. ord. org. solvents.

1-*Me*-3(5)-*Et ester*:  $\text{C}_{14}\text{H}_{14}\text{O}_4\text{N}_2$ . MW, 274. Needles from EtOH. M.p.  $75-6^\circ$ . Sol. ord. org. solvents.

Auwers, Ungemach, *Ber.*, 1933, **66**, 1203.

**3 - Phenylpyrazole - 1 : 4 - dicarboxylic Acid**

$\text{C}_{11}\text{H}_8\text{O}_4\text{N}_2$

MW, 232

4-*Et ester*:  $\text{C}_{13}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 260. Cryst. from EtOH. M.p.  $85-6^\circ$ . *Anilide*: cryst. from ligroin. M.p.  $136-7^\circ$ . Sol. ord. org. solvents.

### 3(5)-Phenylpyrazole-1 : 5(3)-dicarboxylic Acid

*Di-Et ester* :  $C_{15}H_{16}O_4N_2$ . MW, 288. Needles from ligroin. M.p. 57.5–58.5°. Sol. ord. org. solvents. Spar. sol. ligroin.

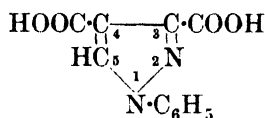
Auwers, Ungemach, *Ber.*, 1933, **66**, 1207.

### 3(5)-Phenylpyrazole-1 : 5(3)-dicarboxylic Acid.

*1-Me-5(3)-Et ester* :  $C_{14}H_{14}O_4N_2$ . MW, 274. Needles from ligroin. M.p. 80–1°. Sol. ord. org. solvents.

Auwers, Dietrich, *J. prakt. Chem.*, 1934, **139**, 93.

### 1 - Phenylpyrazole - 3 : 4 - dicarboxylic Acid



$C_{11}H_8O_4N_2$  MW, 232

Plates from  $H_2O$ . M.p. 234° decomp. Spar. sol. cold  $H_2O$ ,  $Et_2O$ .

*Di-Me ester* :  $C_{13}H_{12}O_4N_2$ . MW, 260. Tablets. M.p. 97–8°.

Balbiano, *Gazz. chim. ital.*, 1898, **28**, i, 385.

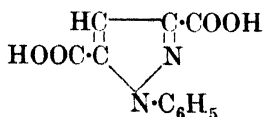
### 5 - Phenylpyrazole - 3 : 4 - dicarboxylic Acid.

Cryst. M.p. 235° decomp. Very sol. hot  $H_2O$ ,  $EtOH$ . Mod. sol.  $Et_2O$ . Resorcinol  $\rightarrow$  fluorescein reaction.

Buchner, *Ber.*, 1894, **27**, 3247.

Sjollema, *Ann.*, 1894, **279**, 252.

### 1 - Phenylpyrazole - 3 : 5 - dicarboxylic Acid



$C_{11}H_8O_4N_2$  MW, 232

Plates from  $EtOH$ . M.p. 266° decomp. to 1-phenylpyrazole-3-carboxylic acid. Sol.  $Et_2O$ . Spar. sol. cold  $H_2O$ ,  $CHCl_3$ , ligroin,  $C_6H_6$ .

*Di-NH<sub>4</sub> salt* : plates. M.p. 210–15° decomp. Very sol.  $H_2O$ .

*Di-Me ester* :  $C_{13}H_{12}O_4N_2$ . MW, 260. M.p. 127–8°.

*Diamide* :  $C_{11}H_{10}O_2N_4$ . MW, 230. Plates from  $EtOH$ . M.p. 190°. Very sol.  $EtOH$ .

Claisen, Roosen, *Ann.*, 1894, **278**, 286.

### 4 - Phenylpyrazole - 3 : 5 - dicarboxylic Acid.

Needles +  $2H_2O$  from  $H_2O$ . Loses  $H_2O$  at 130°. M.p. 243–6°. Dist.  $\rightarrow$  4-phenylpyrazole.

445

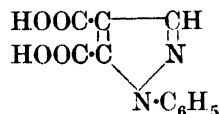
### 4-Phenylpyrazoline

*Me-Et ester* :  $C_{14}H_{14}O_4N_2$ . MW, 274. Rhombohedra or needles from  $Et_2O$ . M.p. 104–5°.

*Di-Et ester* :  $C_{15}H_{16}O_4N_2$ . MW, 288. Needles from  $EtOH$ . Aq. M.p. 96°.

Behaghel, Buchner, *Ber.*, 1902, **35**, 34.

### 1 - Phenylpyrazole - 4 : 5 - dicarboxylic Acid



$C_{11}H_8O_4N_2$  MW, 232

Prisms and plates from  $H_2O$ . M.p. 216°. Sol.  $EtOH$ . Very sol. hot  $H_2O$ . Spar. sol. cold  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ . Dist.  $\rightarrow$  1-phenylpyrazole-4-carboxylic acid. Resorcinol  $\rightarrow$  greenish-red fluorescence.

*Ba salt* : needles from  $H_2O$ . Spar. sol. cold  $H_2O$ .

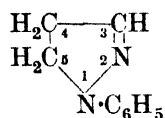
*Di-Me ester* :  $C_{13}H_{12}O_4N_2$ . MW, 260. Needles from  $MeOH$ . M.p. 75–6°.

*Diamide* :  $C_{11}H_{10}O_2N_4$ . MW, 230. Needles from  $H_2O$ . M.p. 253–5°.

*Dianilide* : plates. M.p. 205–6°. Sol.  $EtOH$ ,  $AcOEt$ . Spar. sol.  $Et_2O$ , ligroin.

Claisen, *Ann.*, 1897, **295**, 315.

### 1-Phenylpyrazoline



$C_9H_{10}N_2$  MW, 146

Plates from ligroin. M.p. 51–2°. B.p. 273–4°/754 mm. Sol. hot  $H_2O$ ,  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ . Slightly basic. Volatile in steam from slightly acid sol.

Michaelis, Lampe, *Ann.*, 1893, **274**, 319.

### 3-Phenylpyrazoline.

M.p. 44–5°. B.p. 164°/17 mm. Spar. sol. pet. ether.

*Picrate* : cryst. from  $MeOH$ . M.p. 142–3°.

*Nitroso deriv.* : m.p. 152.5–153.5°.

Auwers, Heimke, *Ann.*, 1927, **458**, 207.

### 4-Phenylpyrazoline.

Oil. Oxidises in air to 4-phenylpyrazole.

*B.HCl* : cryst. from  $EtOH$ . M.p. 162°. Sol.  $H_2O$ ,  $EtOH$ . Insol.  $Et_2O$ .

$B_2(COOH)_2$  : cryst. from  $EtOH$ . M.p. 120°.

$B_2H_2PtCl_6$  : m.p. 116–17°.

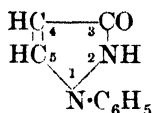
Oliveri, Mandala, *Gazz. chim. ital.*, 1910, **40**, i, 117.

Buchner, Dessauer, *Ber.*, 1903, **36**, 3777.

**5-Phenylpyrazoline.**

Colourless oil. B.p. 153°/15 mm.  $D_4^{20}$  1.1068.  
Picrate: m.p. 117–18°.

Auwers, Heimke, *Ann.*, 1927, **458**, 208.

**1-Phenylpyrazolone-3**

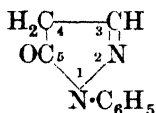
$C_9H_8ON_2$  MW, 160

Needles from  $H_2O$ . M.p. 154°. Sol. hot EtOH,  $CHCl_3$ ,  $C_6H_6$ , conc. HCl, alkalis. Spar. sol. hot  $H_2O$ .

$B, HCl$ : needles. M.p. 111°.

*N*-Acetyl: needles from ligroin. M.p. 62–3°.

Harries, Loth, *Ber.*, 1896, **29**, 519.

**1-Phenylpyrazolone-5**

$C_9H_8ON_2$  MW, 160

Prisms and plates from AcOEt. M.p. 118–19°. Sol. acids, alkalis.

$B, HCl$ : needles. M.p. 165°.

*Isonitroso deriv.*: orange-yellow needles from AcOH.Aq. M.p. 160° decomp.

Claisen, Haase, *Ber.*, 1895, **28**, 38.

**3-Phenylpyrazolone-5.**

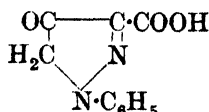
Plates from EtOH. M.p. 236° decomp. Insol. cold EtOH,  $Et_2O$ ,  $C_6H_6$ .

$B, HCl$ : needles. M.p. 196°.

*N*-Acetyl: plates from EtOH. M.p. 122°. Spar. sol. cold EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Isonitroso deriv.*: yellow needles from  $H_2O$ . M.p. 188°. Sol. EtOH,  $Et_2O$ , alkalis.

Rothenburg, *J. prakt. Chem.*, 1895, **52**, 23.

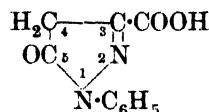
**1-Phenyl-4-pyrazolone-3-carboxylic Acid (1-Phenyl-isopyrazolone-3-carboxylic acid)**

$C_{10}H_8O_3N_2$  MW, 204

*Me ester*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Needles from MeOH. M.p. 85–7°.  $FeCl_3$  → blue col.

*Et ester*:  $C_{13}H_{12}O_3N_2$ . MW, 232. Pale yellow needles from EtOH. M.p. 258–60°.  $FeCl_3$  → blue col.

Favrel, *Compt. rend.*, 1913, **156**, 1912.

**1-Phenyl-5-pyrazolone-3-carboxylic Acid**

$C_{10}H_8O_3N_2$  MW, 204

Small needles from EtOH or hot  $H_2O$ . Loses  $CO_2$  at 230°, m.p. 261°. Sol. EtOH. Insol.  $H_2O$ ,  $Et_2O$ .  $FeCl_3$  → blue col.

*Me ester*:  $C_{11}H_{10}O_3N_2$ . MW, 218. Cryst. from MeOH. M.p. 197°.

*Et ester*:  $C_{13}H_{12}O_3N_2$ . MW, 232. Prisms from EtOH. M.p. 180–2°.  $FeCl_3$  → blue col.

*Et ether*: plates from  $H_2O$ . M.p. 152–3°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol. cold  $H_2O$ .

*Et ester*: needles from EtOH.Aq. M.p. 83–4°.

Leighton, *J. Am. Chem. Soc.*, 1898, **20**, 679.

**1-Phenyl-5-pyrazolone-4-carboxylic Acid.**

M.p. 91–2° decomp. Sol. EtOH,  $Me_2CO$ .

*Et ester*: needles from EtOH.Aq. M.p. 117–18°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ , alkalis.  $FeCl_3$  → dark red col.

Ruhemann, *J. Chem. Soc.*, 1893, **63**, 878.

**2-Phenyl-5-pyrazolone-4-carboxylic Acid.**

Cryst. from EtOH. M.p. 216° decomp. Dist. → 2-phenylpyrazolone-5.

Michaelis, Remy, *Ber.*, 1907, **40**, 1020.

**3-Phenylpyridazine (3-Phenyl-1:2-diazine)**

$C_{10}H_8N_2$  MW, 156

Needles from  $H_2O$  or ligroin. M.p. 102–3°. B.p. 330–2°. Sol. EtOH,  $Et_2O$ . Sublimes.

$B, MeI$ : yellow needles from MeOH– $Et_2O$ . M.p. 179°. Sol. MeOH.

$B, H_2AuCl_4$ : yellow needles. M.p. 159°.

Picrate: cryst. from EtOH. M.p. 127°.

Gabriel, Colman, *Ber.*, 1899, **32**, 401.

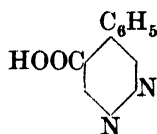
**4-Phenylpyridazine (4-Phenyl-1:2-diazine).**

Cryst. from ligroin. M.p. 86–86.5°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , acids. Spar. sol. ligroin.

$B_2, H_2PtCl_6, H_2O$ : cryst. M.p. 295–300° decomp. Spar. sol.  $H_2O$ , EtOH.

Stoermer, Fincke, *Ber.*, 1909, **42**, 3130.

## 4-Phenylpyridazine-5-carboxylic Acid

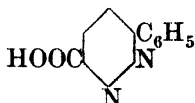
 $C_{11}H_8O_2N_2$ 

MW, 200

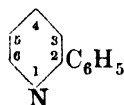
Slightly brownish crystals. M.p. 220–1° decomp. Mod. sol. hot EtOH. Spar. sol. hot H<sub>2</sub>O. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Heat with BaCO<sub>3</sub> in vacuo → 4-phenylpyridazine.

See previous reference.

## 3-Phenylpyridazine-6-carboxylic Acid

 $C_{11}H_8O_2N_2$ 

MW, 200

Needles from H<sub>2</sub>O. M.p. 130–1°.Paal, Dencks, *Ber.*, 1903, 36, 494.2-Phenylpyridine ( $\alpha$ -Pyridylbenzene) $C_{11}H_9N$ 

MW, 155

Oil. B.p. 268–9°. Misc. with EtOH, Et<sub>2</sub>O, but not H<sub>2</sub>O. Difficultly volatile in steam. Neutral KMnO<sub>4</sub> → benzoic acid. Acid KMnO<sub>4</sub> → picolinic acid.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>·2H<sub>2</sub>O: orange-yellow needles. M.p. 204°. Insol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

Picrate: pale yellow needles. M.p. 175°. Very sol. hot EtOH.

Ziegler, Zeiser, *Ber.*, 1930, 63, 1851.Bergstrom, McAllister, *J. Am. Chem. Soc.*, 1930, 52, 2847.3-Phenylpyridine ( $\beta$ -Pyridylbenzene).

Pale yellow oil. B.p. 269–70°/749 mm. Immiscible with H<sub>2</sub>O. Sol. EtOH, Et<sub>2</sub>O, dil. acids. Difficultly volatile in steam. KMnO<sub>4</sub> → nicotinic acid.

Picrate: pale yellow needles from EtOH. M.p. 162–3°.

Skraup, Cobenzl, *Monatsh.*, 1883, 4, 456.4-Phenylpyridine ( $\gamma$ -Pyridylbenzene).

Plates from H<sub>2</sub>O. M.p. 77–8°. B.p. 274–5°. Mod. sol. hot H<sub>2</sub>O. KMnO<sub>4</sub> → isonicotinic acid. Na + EtOH → 4-phenylpiperidine.

B, MeI: nearly colourless cryst.

B<sub>2</sub>H<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>: orange needles from H<sub>2</sub>O. M.p. 155° decomp.

Picrate: yellow needles. M.p. 197·5°.

Overhoff, Tilman, *Rec. trav. chim.*, 1929, 48, 993.

## 6-Phenylpyridine-2-carboxylic Acid.

See 6-Phenylpicolinic Acid.

## 6-Phenylpyridine-3-carboxylic Acid.

See 6-Phenylnicotinic Acid.

## Phenylpyridine-dicarboxylic Acid.

See Phenylcinchomeronic Acid, Phenylidnicotinic Acid, and Phenylquinolinic Acid.

1-Phenyl- $\alpha$ -pyridone $C_{11}H_9ON$ 

MW, 171

Cryst. from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 128°. B.p. above 300°.

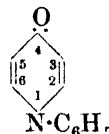
Tschitschibabin, Geletzki, *Ber.*, 1924, 57, 1159.

6-Phenyl- $\alpha$ -pyridone.

Yellow scales from C<sub>6</sub>H<sub>6</sub>. M.p. 197°. Sol. EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, Et<sub>2</sub>O. Zn dust dist. → 2-phenylpyridine. PCl<sub>3</sub> → 6-chloro-2-phenylpyridine.

B, HCl: pale yellow needles. M.p. 104°.

Leben, *Ber.*, 1896, 29, 1678.

1-Phenyl- $\gamma$ -pyridone $C_{11}H_9ON$ 

MW, 171

Needles + 2H<sub>2</sub>O from H<sub>2</sub>O, m.p. anhyd. 104–5°: cryst. from C<sub>6</sub>H<sub>6</sub>, m.p. 116°. Very sol. warm H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O.

B, MeI: rhomboids. M.p. 146°.

Picrate: yellow leaflets from EtOH. M.p. 190°.

Borsche, Bonacker, *Ber.*, 1921, 54, 2682.

6-Phenyl- $\gamma$ -pyridone.

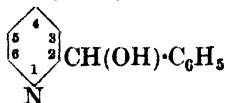
Cryst. + ½H<sub>2</sub>O from H<sub>2</sub>O. M.p. 155°.

Borsch, Peter, *Ann.*, 1927, 453, 158.

## Phenyl-2-pyridylamine.

See 2-Anilinopyridine.

Phenyl-2-pyridylcarbinol ( $\alpha$ -[ $\alpha$ -Pyridyl]-benzyl alcohol, 2- $\alpha$ -hydroxybenzylpyridine)

 $C_{12}H_{11}ON$ 

MW, 185



Cryst. from  $C_6H_6$ -pet. ether. M.p.  $82^\circ$ . Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , dil. min. acids. Insol.  $H_2O$ , pet. ether.  $KMnO_4 \rightarrow$  phenyl 2-pyridyl ketone.

$B_2H_2PtCl_6$ : orange-red plates from  $H_2O$ . M.p.  $197^\circ$  decomp. Spar. sol. cold  $H_2O$ .

Tschitschibabin, *Ber.*, 1904, **37**, 1371.

**Phenyl-4-pyridylcarbinol** ( $\alpha$ -[ $\gamma$ -Pyridyl]-benzyl alcohol, 4- $\alpha$ -hydroxybenzylpyridine).

Cryst. from  $C_6H_6$  or AcOEt. M.p.  $126^\circ$ . Very sol. EtOH, min. acids. Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ , pet. ether.  $KMnO_4 \rightarrow$  phenyl 4-pyridyl ketone.

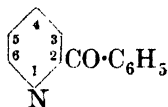
$B_2H_2PtCl_6$ : orange-red scales from  $H_2O$ . M.p.  $205^\circ$ . Spar. sol. cold  $H_2O$ .

See previous reference.

**Phenylpyridylethane.**

See Phenylethylpyridine.

**Phenyl 2-pyridyl Ketone** (2-Benzoylpyridine)



$C_{12}H_9ON$  MW, 183

Oil. B.p.  $317^\circ$ ,  $170-2^\circ/10$  mm.  $D_0^{20}$  1.1553. Red.  $\rightarrow$  phenyl-2-pyridylcarbinol.

$B.HCl$ : cryst. from  $Me_2CO$ . M.p.  $126-8^\circ$ .

$B_2H_2PtCl_6$ : cryst. from dil. HCl. M.p.  $193^\circ$  decomp.

*Oxime*: two forms. (i) Pale yellow prisms. M.p.  $150-2^\circ$ . (ii) Cryst. M.p.  $165-7^\circ$ .

*Phenylhydrazone*: yellow cryst. from EtOH. M.p.  $136-7^\circ$ . Spar. sol. cold EtOH.

*Picrate*: cryst. from EtOH or  $Me_2CO$ . M.p.  $130^\circ$ . Sol. MeOH, AcOEt. Spar. sol.  $C_6H_6$ .

Crook, McElvain, *J. Am. Chem. Soc.*, 1930, **52**, 4006.

Tschugajew, *Ber.*, 1906, **39**, 3387.

Tschitschibabin, *Chem. Zentr.*, 1902, **I**, 206.

**Phenyl 3-pyridyl Ketone** (3-Benzoylpyridine).

Cryst. M.p.  $42^\circ$ . B.p.  $318-19^\circ$ . Ox.  $\rightarrow$  nicotinic acid. Red.  $\rightarrow$  3-benzylpyridine.

$B.HCl$ : needles. M.p.  $160-2^\circ$ .

$B_2H_2PtCl_6$ : cryst. from dil. HCl. M.p.  $245^\circ$  decomp.

*Oxime*: two forms. (i) M.p.  $141-3^\circ$ . Sol. hot EtOH. Spar. sol.  $H_2O$ . (ii) Prisms from  $C_6H_6$ . M.p.  $162-3^\circ$ . Sol. EtOH. Spar. sol.  $Et_2O$ . Insol.  $H_2O$ . Sol. in dil. HCl, pptd. with  $Na_2CO_3 \rightarrow$  (i).

*Phenylhydrazone*: m.p.  $143-5^\circ$ .

*Picrate*: clusters of needles from EtOH. M.p.  $161^\circ$ .

La Forge, *J. Am. Chem. Soc.*, 1928, **50**, 2486.

Tschitschibabin, *Ber.*, 1903, **36**, 2711.

**Phenyl 4-pyridyl Ketone** (4-Benzoylpyridine).

Plates from  $H_2O$ , needles from pet. ether. M.p.  $71.5-72.5^\circ$ . B.p.  $313.5-314^\circ/742$  mm. Very sol. EtOH,  $Et_2O$ . Spar. sol. hot  $H_2O$ . Red.  $\rightarrow$  phenyl-4-pyridylcarbinol.

$B.HCl$ : cryst. from EtOH- $Et_2O$ . M.p.  $195-7^\circ$ .

*Oxime*: two forms. (i) Monoclinic prisms from EtOH. M.p.  $176-7^\circ$  decomp. (ii) Cryst. from EtOH. M.p.  $152-5^\circ$ .

*Phenylhydrazone*: yellow cryst. from EtOH. M.p.  $181-2^\circ$ .

*Picrate*: monoclinic cryst. from EtOH. M.p.  $159-60^\circ$ .

Crook, McElvain, *J. Am. Chem. Soc.*, 1930, **52**, 4006.

Tschitschibabin, *Chem. Zentr.*, 1902, **I**, 206.

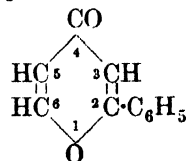
**Phenylpyridylmethane.**

See Benzylpyridine.

**6-Phenyl- $\alpha$ -pyrone.**

See 6-Phenylcoumalin.

**2-Phenyl- $\gamma$ -pyrone**

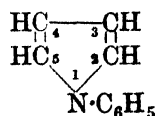


$C_{11}H_8O_2$  MW, 172

Needles from  $H_2O$ . M.p.  $104^\circ$ .

Borsche, Péter, *Ann.*, 1927, **453**, 158.

**1-Phenylpyrrole**



$C_{10}H_9N$  MW, 143

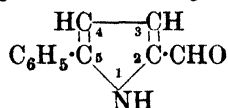
Scales with camphor-like odour. M.p.  $62^\circ$ . B.p.  $234^\circ$ ,  $140^\circ/38$  mm. Very sol. pet. ether. Mod. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Insol.  $H_2O$ . Readily volatile in steam. Sublimes. Heat of comb. C, 1283.1 Cal. Turns red in air. Unaffected by acids and alkalis. EtOH-HCl  $\rightarrow$  "pine-chip" violet. Dist. through glowing tube  $\rightarrow$  2-phenylpyrrole.

Kötnitz, *J. prakt. Chem.*, 1872, **6**, 448.

**2-Phenylpyrrole.**

Plates from EtOH. M.p. 129°. B.p. 271-2°/726 mm. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. Sublimes. Readily volatile in steam.

Pictet, Crépieux, *Ber.*, 1895, **28**, 1905.

**5-Phenylpyrrole-2-aldehyde**

C<sub>11</sub>H<sub>9</sub>ON

MW, 171

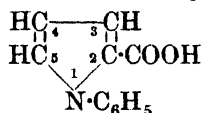
Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 138°.

*Semicarbazone*: greenish plates from EtOH.Aq. M.p. 190°.

*p*-Nitrophenylhydrazone: dark red needles from EtOH. M.p. 222°.

*Azine*: yellow cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 240°.

Plancher, Rossi, Ghigi, *Gazz. chim. ital.*, 1929, **59**, 347.

**1-Phenylpyrrole-2-carboxylic Acid**

C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 187

Plates or needles from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 166° decomp. to 1-phenylpyrrole. Very sol. EtOH, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>. Sol. Et<sub>2</sub>O. Insol. cold H<sub>2</sub>O, pet. ether. Loses CO<sub>2</sub> in boiling water.

*Me ester*: C<sub>12</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 201. Plates from EtOH.Aq. M.p. 88°. B.p. 282°. Insol. H<sub>2</sub>O.

*Et ester*: C<sub>13</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 215. B.p. 289°.

*Anilide*: needles from EtOH.Aq. M.p. 136°.

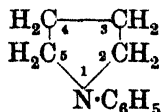
Pictet, Steinmann, *Ber.*, 1902, **35**, 2530.

**2-Phenylpyrrole-3-carboxylic Acid.**

Cubes. M.p. 192-3° decomp.

*Et ester*: prisms. M.p. 19°. B.p. 194-5°/6 mm.

Kondo, Suzuki, *Chem. Zentr.*, 1927, **II**, 1029.

**1-Phenylpyrrolidine**

C<sub>10</sub>H<sub>13</sub>N

MW, 147

Oil. B.p. 110-16°/9 mm. D<sub>25</sub> 1.0260. n<sub>D</sub><sup>25</sup> 1.5803.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: yellow needles from EtOH-HCl. M.p. 174-5° decomp.

Dict. of Org. Comp.—III.

B<sub>2</sub>(COOH)<sub>2</sub>.H<sub>2</sub>O: cryst. from Me<sub>2</sub>CO-pet. ether. M.p. 156°.

*Picrate*: m.p. 115-16°.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 715.

Craig, Hixon, *J. Am. Chem. Soc.*, 1930, **52**, 807.

**2-Phenylpyrrolidine.**

Oil. B.p. 236-8°. Spar. sol. H<sub>2</sub>O giving alk. sol. Absorbs CO<sub>2</sub> from the air.

B<sub>2</sub>HAuCl<sub>4</sub>: yellow prisms. M.p. 110°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: prisms. M.p. 187-8°.

*Picrate*: plates. M.p. 148-9°.

La Forge, *J. Am. Chem. Soc.*, 1928, **50**, 2477.

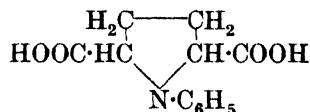
Gabriel, Colman, *Ber.*, 1908, **41**, 520.

**3-Phenylpyrrolidine.**

Oil. B.p. 120-2°/12 mm. Spar. sol. H<sub>2</sub>O. Slowly absorbs CO<sub>2</sub> from the air.

*Picrate*: cryst. from EtOH. M.p. 166°.

Späth, Breusch, *Monatsh.*, 1928, **50**, 352.

**1-Phenylpyrrolidine-2:5-dicarboxylic Acid**

C<sub>12</sub>H<sub>13</sub>O<sub>4</sub>N

MW, 235

Needles from Me<sub>2</sub>CO-pet. ether. Decomp. at 252°. Very sol. cold EtOH, hot Me<sub>2</sub>CO. Spar. sol. cold Et<sub>2</sub>O, H<sub>2</sub>O. Insol. CHCl<sub>3</sub>, pet. ether, C<sub>6</sub>H<sub>6</sub>. Decolourises acid or alk. KMnO<sub>4</sub>.

*Ba salt*, 8H<sub>2</sub>O: needles from EtOH.Aq. Very sol. H<sub>2</sub>O. Insol. abs. EtOH.

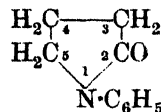
*Di-Me ester*: C<sub>14</sub>H<sub>17</sub>O<sub>4</sub>N. MW, 263. Needles from pet. ether. M.p. 88°. B.p. 225-30°/32 mm. Very sol. hot EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. cold H<sub>2</sub>O, cold EtOH, pet. ether.

*Di-Et ester*: C<sub>16</sub>H<sub>21</sub>O<sub>4</sub>N. MW, 291. Yellow oil. B.p. 227-8°/30 mm. Immiscible with H<sub>2</sub>O.

*Monoanilide*: needles from CHCl<sub>3</sub>-C<sub>6</sub>H<sub>6</sub>. Decomp. at 184°. Very sol. EtOH, Me<sub>2</sub>CO. Spar. sol. cold H<sub>2</sub>O, pet. ether, C<sub>6</sub>H<sub>6</sub>.

Le Sueur, Haas, *J. Chem. Soc.*, 1910, **97**, 177.

Le Sueur, *J. Chem. Soc.*, 1909, **95**, 276.

**1-Phenylpyrrolidone-2 (N-Phenyl-γ-butyrolactam)**

C<sub>10</sub>H<sub>11</sub>ON

MW, 161  
29

Needles from EtOH.Aq. or C<sub>6</sub>H<sub>6</sub>-pet. ether, plates from H<sub>2</sub>O. M.p. 68-9°. B.p. 180-2°/11 mm.

Lipp, Caspers, *Ber.*, 1925, **58**, 1013.

**4-Phenylpyrrolidone-2** (2-Phenyl-γ-butyrolactam).

Hexagonal prisms from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 60°.

N-Acetyl: hexagonal plates from EtOH. M.p. 63°.

N-Benzoyl: prisms from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 145°.

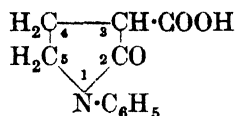
Jackson, Kenner, *J. Chem. Soc.*, 1928, 1659.

**5-Phenylpyrrolidone-2** (3-Phenyl-γ-butyrolactam).

Needles. M.p. 91°.

Köhl, *Ber.*, 1903, **36**, 174.

**1-Phenyl-2-pyrrolidone-3-carboxylic Acid**



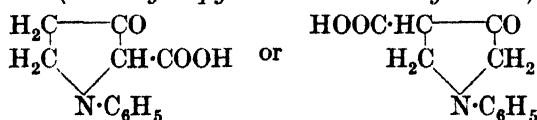
C<sub>11</sub>H<sub>11</sub>O<sub>3</sub>N

MW, 205

M.p. 64-5°.

Küster, Grassner, *Z. physiol. Chem.*, 1925, **145**, 45.

**1-Phenyl-3-pyrrolidone-2-carboxylic Acid** (1-Phenyl-3-pyrrolidone-4-carboxylic acid)



C<sub>11</sub>H<sub>11</sub>O<sub>3</sub>N

MW, 205

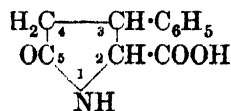
Cryst. from H<sub>2</sub>O. M.p. 143-4°. Losses CO<sub>2</sub> at 170-80°. FeCl<sub>3</sub> → no col.

Et ester: C<sub>13</sub>H<sub>15</sub>O<sub>3</sub>N. MW, 233. Needles from EtOH, plates from EtOH.Aq. M.p. 69-70°. FeCl<sub>3</sub> → dark violet col.

Phenylhydrazone: m.p. 160-1°.

de Moulpied, *J. Chem. Soc.*, 1905, **87**, 442.

**3-Phenyl-5-pyrrolidone-2-carboxylic Acid**



C<sub>11</sub>H<sub>11</sub>O<sub>3</sub>N

MW, 205

Needles and prisms from EtOH. M.p. 196.5-197.5°.

Harington, *J. Biol. Chem.*, 1925, **64**, 29.

**1-Phenyl-5-pyrrolidone-3-carboxylic Acid.**

Needles from H<sub>2</sub>O, plates from EtOH.Aq. M.p. 190° slight decomp. Insol. dil. HCl.

Ag salt: needles from H<sub>2</sub>O.

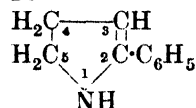
Ba salt: very sol. H<sub>2</sub>O.

Chloride: C<sub>11</sub>H<sub>10</sub>O<sub>2</sub>NCl. MW, 223.5. Cryst. Easily decomp. by H<sub>2</sub>O.

Anilide: plates from EtOH.Aq. M.p. 185°. Very sol. Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Sublimes.

Anschütz, Reuter, *Ann.*, 1889, **254**, 141.

**2-Phenyl-2-pyrroline**



C<sub>10</sub>H<sub>11</sub>N

MW, 145

Cryst. with strong ammoniacal odour and bitter taste. M.p. 44-5°. B.p. 249°/752 mm. Spar. sol. hot H<sub>2</sub>O. Aq. sol. reacts alkaline. Red. → 2-phenylpyrrolidine.

B.HCl: plates from Me<sub>2</sub>CO or EtOH-Et<sub>2</sub>O. M.p. 210° (206-7°).

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: orange plates or needles from dil. HCl. M.p. 220° decomp. (darkens at 200°).

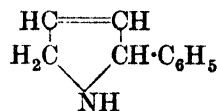
Picrate: cryst. from MeOH. M.p. 198°.

Gabriel, Colman, *Ber.*, 1908, **41**, 517.

Sonn, Podschus, Schützler, Stephani, *Ber.*, 1935, **68**, 150.

Craig, Bulbrook, Hixon, *J. Am. Chem. Soc.*, 1931, **53**, 1833.

**2-Phenyl-3-pyrroline**



C<sub>10</sub>H<sub>11</sub>N

MW, 145

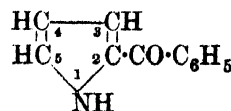
M.p. 45°. B.p. 118°/11 mm.

B.HCl: cryst. from EtOH.Aq. M.p. 240°.

Lipp, Seeles, *Ber.*, 1929, **62**, 2458.

Wohl, *Ber.*, 1901, **34**, 1922.

**Phenyl 2-pyrrolyl Ketone** (2-Benzoylpyrrole)



C<sub>11</sub>H<sub>9</sub>ON

MW, 117

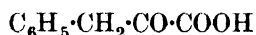
Needles, plates or prisms from hot  $\text{H}_2\text{O}$ ,  $\text{EtOH.Aq.}$ , or pet. ether. M.p.  $79^\circ$ . B.p.  $305^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ . Sol. pet. ether. Insol. cold  $\text{H}_2\text{O}$ . Stable to boiling  $\text{KOH}$ .

*Oxime*: needles from  $\text{EtOH.Aq.}$  M.p.  $147^\circ$ .

Oddo, Dainotti, *Gazz. chim. ital.*, 1912, **42**, i, 730.

Pictet, *Ber.*, 1904, **37**, 2797.

**Phenylpyruvic Acid** ( $\alpha$ -Ketohydrocinnamic acid)



$\text{C}_9\text{H}_8\text{O}_3$

MW, 164

Plates from  $\text{CHCl}_3$ . M.p.  $157^\circ$  ( $153\text{--}4^\circ$ ) decomp. Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Sol. hot  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. boiling  $\text{H}_2\text{O}$ . Insol. cold ligroin. Oxidises in air. Freshly prepared sol. in  $\text{K}_2\text{CO}_3$  decolourises  $\text{KMnO}_4$ . Reduces boiling Fehling's and Nessler's. Salts react with semicarbazide but free acid does not.  $\text{Et}_2\text{O}$  sol.  $\rightarrow$  green ring with  $\text{FeCl}_3$  sol.  $\text{Ac}_2\text{O} \rightarrow$  1-acetoxycinnamic acid. Red.  $\rightarrow$  benzylglycollic acid.

*Na salt*,  $\text{H}_2\text{O}$ : insol.  $\text{EtOH}$ . Stable in air.  $\text{H}_2\text{O}$  of cryst. remains at  $100^\circ$ .

*Me ester*:  $\text{C}_{10}\text{H}_{10}\text{O}_3$ . MW, 178. Needles. M.p.  $75^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . Oxidises in air  $\rightarrow$   $\text{C}_6\text{H}_5\cdot\text{CHO}$ . *Semicarbazone*: m.p.  $196^\circ$ .

*Et ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_3$ . MW, 192. Needles. M.p.  $45^\circ$ . B.p.  $154\cdot5^\circ/15\text{ mm.}$ ,  $117\text{--}21^\circ/2\cdot8\text{ mm.}$  Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . Oxidises in air  $\rightarrow$   $\text{C}_6\text{H}_5\cdot\text{CHO}$ .  $\text{FeCl}_3 \rightarrow$  violet col.  $\text{Ac}_2\text{O} \rightarrow$  ethyl 1-acetoxycinnamic ester. Can be separated into 3 forms, 2 enolic *cis-trans* isomerides and one ketonic:—

$\alpha$ . Needles from ligroin. M.p.  $52^\circ$ .  $\beta$ . B.p.  $152^\circ/15\text{ mm.}$   $\gamma$ . M.p.  $79^\circ$ .  $\alpha$  and  $\beta$  with  $\text{FeCl}_3 \rightarrow$  green col.  $\gamma$  Gives no col.  $\alpha$  and  $\beta$  give same phenylurethane of ethyl 1-hydroxycinnamic ester. All give same acetate and benzoate of 1-OH-cinnamic ester, and same ketonic derivatives. *Oxime*:  $\text{C}_{11}\text{H}_{13}\text{O}_3\text{N}$ . MW, 207. Prisms or needles from ligroin. M.p.  $57\text{--}8^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Mod. sol. ligroin. Spar. sol.  $\text{H}_2\text{O}$ . *Semicarbazone*: plates from  $\text{EtOH.Aq.}$  M.p.  $159\text{--}60^\circ$  ( $167^\circ$ ). *Phenylhydrazones*: m.p.  $89^\circ$ . *p-Tolylhydrazones*: orange prisms. M.p.  $72^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . *p-Nitrophenylhydrazones*: yellow cryst. from  $\text{AcOH}$ . M.p.  $181^\circ$ .

*Amide*:  $\text{C}_9\text{H}_9\text{O}_2\text{N}$ . MW, 163. M.p.  $190^\circ$ . Sol. hot  $\text{EtOH}$ . Insol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . *Oxime*: needles from  $\text{H}_2\text{O}$ . M.p.  $147^\circ$ .

*Anilide*: m.p.  $126^\circ$ .

*Oxime*:  $\text{C}_9\text{H}_9\text{O}_3\text{N}$ . MW, 179. Needles from  $\text{EtOH.Aq.}$ , or  $\text{H}_2\text{O}$ . M.p.  $159^\circ$  ( $153\text{--}67^\circ$ ) decomp. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*Semicarbazone*: m.p.  $180^\circ$  decomp.

*p-Nitrophenylhydrazones*: cryst. from  $\text{EtOH.Aq.}$  M.p.  $187\text{--}8^\circ$  decomp.

*p-Tolylhydrazones*: yellow felted needles from  $\text{EtOH}$ , prisms from  $\text{Me}_2\text{CO}$ . M.p.  $158^\circ$  decomp.

Erlenmeyer, Arbenz, *Ann.*, 1904, **333**, 228.

Bougault, *Compt. rend.*, 1916, **162**, 761.

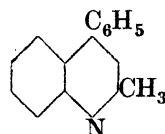
Gault, Weick, *Bull. soc. chim.*, 1922, **31**, 867.

Feist, Rauterberg, *Ber.*, 1922, **55**, 3702.

Walker, *J. Chem. Soc.*, 1925, 1862.

Kon, Watson, *J. Chem. Soc.*, 1932, 7.

**4-Phenylquinaldine** (2-Methyl-4-phenylquinoline)



$\text{C}_{16}\text{H}_{13}\text{N}$

MW, 219

Plates from  $\text{EtOH}$ . M.p.  $98\text{--}9^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin, pet. ether. Sol. dil. acids with blue fluor.

*Hydrochloride*: needles from  $\text{EtOH}$ . M.p.  $219^\circ$ .

*Sulphate*: yellow needles from  $\text{H}_2\text{O}$ . M.p.  $235^\circ$ .

*ZnCl<sub>2</sub> double salt*: needles. M.p.  $197^\circ$ .

*CdCl<sub>2</sub> double salt*: needles. M.p.  $208\text{--}10^\circ$ .

*HgCl<sub>2</sub> double salt*: needles from dil.  $\text{HCl}$ . M.p.  $202^\circ$ .

*Chloroplatinate*: m.p.  $235^\circ$ .

*Picrate*: yellow needles from  $\text{EtOH}$ . M.p.  $205\text{--}6^\circ$ . Insol.  $\text{Et}_2\text{O}$ .

*Methiodide*: m.p.  $205^\circ$ .

Spallino, Salimei, *Gazz. chim. ital.*, 1912, **42**, i, 608.

Fischer, *J. prakt. Chem.*, 1918, **98**, 226.

Beyer, *J. prakt. Chem.*, 1886, **33**, 421.

**4-Phenylquinaldinic Acid**.

See 4-Phenylquinoline-2-carboxylic Acid.

**2-Phenylquinazoline**



$\text{C}_{14}\text{H}_{10}\text{N}_2$

MW, 206

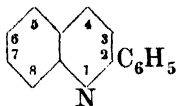
Needles from  $\text{EtOH}$ . M.p.  $101^\circ$ . Does not boil below  $300^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ .

Bischler, Lang, *Ber.*, 1895, **28**, 288.

**4-Phenylquinazoline.**

*Picrate*: yellow leaflets from EtOH. M.p. 178°.

Bischler, Barad, *Ber.*, 1892, 25, 3093.

**2-Phenylquinoline**

$C_{15}H_{11}N$

MW, 205

Needles from EtOH.Aq. M.p. 86° (80–1°). B.p. 363°, 310°/187 mm. Sol.  $Et_2O$ ,  $Me_2CO$ ,  $AcOEt$ ,  $C_6H_6$ ,  $CS_2$ , hot EtOH. Spar. sol.  $H_2O$ . Very spar. sol. pet. ether. Triboluminescent.  $KMnO_4$  in dil.  $H_2SO_4 \rightarrow N$ -benzoylanthranilic acid.

*Dichromate*: m.p. 145–8°.

*Ferrichloride*: m.p. 169–70°.

*Hydrochloride*: yellow needles from alc. HCl. M.p. 163–5°.

$B_2, 2HCl, AuCl_3$ : needles from HCl. M.p. about 204°.

$B, HAuCl_4$ : m.p. about 160°.

$B_2, H_2PtCl_6, 2H_2O$ : yellow needles. Decomp. about 210°.

*Picrate*: yellow leaflets from EtOH. M.p. 191–2°.

*Methiodide*: orange-red cryst. from EtOH- $Et_2O$ . M.p. 200°.

*Ethiodide*: yellow prisms. M.p. 195°.

Doebner, v. Miller, *Ber.*, 1883, 16, 1665.

Goldschmidt, *Ber.*, 1895, 28, 986.

Doebner, Gieseke, *Ann.*, 1887, 242, 294.

Le Fèvre, Pearson, *J. Chem. Soc.*, 1932, 2807.

**3-Phenylquinoline.**

Plates from  $Et_2O$ . M.p. 52°. B.p. 205–7°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. volatile in steam.

$B, HCl$ : needles from dil. HCl. M.p. 109°.

*Picrate*: yellow cryst. from EtOH. M.p. 205°.

*Methiodide*: yellow needles from MeOH. M.p. 224°.

*Ethiodide*: m.p. 228°.

Friedländer, Göhring, *Ber.*, 1883, 16, 1836.

Hübner, *Ber.*, 1908, 41, 482.

Warren, *J. Chem. Soc.*, 1936, 1367.

**4-Phenylquinoline.**

Needles from  $Et_2O$  or ligroin. M.p. 61–2°. Sol. EtOH,  $Et_2O$  and most indifferent solvents. Insol.  $H_2O$ . Sol. dil. HCl and dil.  $H_2SO_4$  with blue fluor.

*Hydrochloride*: needles from EtOH- $Et_2O$ . M.p. 96–7°.

*Sulphate*: plates from  $H_2O$ . M.p. 195–6°.

$ZnCl_2$  double salt: needles from dil. HCl. M.p. 115–20°.

$CdCl_2$  double salt: needles. M.p. 120–2°.

$HgCl_2$  double salt: needles. M.p. 197–9°.

$B_2, H_2PtCl_6$ : yellow plates. M.p. 244°.

*Picrate*: yellow needles from EtOH. M.p. 225°.

*Methiodide*: yellow needles from EtOH or  $H_2O$ . M.p. 222° decomp.

Kenner, Statham, *J. Chem. Soc.*, 1935, 301.

Koenigs, Nef, *Ber.*, 1886, 19, 2430; 1887, 20, 622.

Koenigs, Meimberg, *Ber.*, 1895, 28, 1038.

**6-Phenylquinoline.**

Plates from EtOH,  $C_6H_6$  or aniline. M.p. 110–11°. B.p. 260°/77 mm. Sol. EtOH,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ , pet. ether. Very spar. sol.  $H_2O$ . Spar. volatile in steam.

*Methiodide*: yellow prisms from  $H_2O$ . M.p. 194° to a red liq.

La Coste, Sorger, *Ann.*, 1885, 230, 8.

**8-Phenylquinoline.**

Viscous oil with yellowish-green fluor. B.p. 283°/187 mm., 270–6°/80 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Very spar. sol.  $H_2O$ .

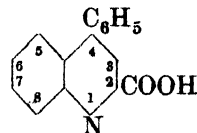
$B_2, H_2Cr_2O_7$ : reddish-yellow leaflets from  $H_2O$ . M.p. 125–6°.

*Picrate*: yellow needles. M.p. about 210°.

*Methiodide*: yellow cryst. M.p. 163°. Insol.  $Et_2O$ .

La Coste, Sorger, *Ann.*, 1885, 230, 39.

Möhlau, Berger, *Ber.*, 1893, 26, 2004.

**4-Phenylquinoline-2-carboxylic Acid**  
(4-Phenylquinaldinic acid)

$C_{16}H_{11}O_2N$

MW, 249

Yellow needles from EtOH.Aq. M.p. 171°. At 180–90°  $\rightarrow$  4-phenylquinoline.

*Chloroplatinate*: yellow needles. M.p. 233–4° decomp.

*Chloride*:  $C_{16}H_{10}ONCl$ . MW, 267.5. Cryst. from ligroin. M.p. 116°  $\rightarrow$  red col.

Koenigs, Jaeglé, *Ber.*, 1895, 28, 1049.

Besthorn, *Ber.*, 1913, 46, 2766.

**2-Phenylquinoline-3-carboxylic Acid.**

Needles. M.p. 226° (230° decomp.). Sol. MeOH, AcOH, PhNO<sub>2</sub>. Spar. sol. C<sub>6</sub>H<sub>6</sub>.

*Me ester*: C<sub>17</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 263. M.p. 84°. B.p. 195°/13 mm.

*Nitrile*: needles from EtOH. M.p. 193-4°.

John, *J. prakt. Chem.*, 1931, **131**, 271.

v. Meyer, *J. prakt. Chem.*, 1914, **90**, 28.

**2-Phenylquinoline-4-carboxylic Acid**

(2-Phenylcinchoninic acid, cinchophene, atophan, quinophan).

Needles from MeOH. M.p. 212-13°. Sol. Et<sub>2</sub>O, hot EtOH, alkalis, warm min. acids. Insol. H<sub>2</sub>O. The acid and several of its derivs. have wide therapeutic use as uric acid eliminants. Oxidised in human organism to 8-hydroxy-2-phenylquinoline-4-carboxylic acid. Dist. with lime → 2-phenylquinoline.

*B,HCl*: lemon-yellow cryst. M.p. 223°.

*B,HBr*: brownish-yellow cryst. M.p. 255°.

*B,HI*: orange cryst. M.p. 243°.

*Me ester*: C<sub>17</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 263. Leaflets. M.p. 61°. Bitter taste.

*Et ester*: C<sub>18</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 277. Cryst. from EtOH. M.p. 51°. Sol. EtOH, Et<sub>2</sub>O. *B,HCl*: m.p. 210-11° decomp. *Picrate*: m.p. 144-5°.

*Propyl ester*: C<sub>19</sub>H<sub>17</sub>O<sub>2</sub>N. MW, 291. M.p. 63-4°.

*Butyl ester*: C<sub>20</sub>H<sub>19</sub>O<sub>2</sub>N. MW, 305. M.p. 56-7°.

*Isobutyl ester*: m.p. 39-40°.

*Isomyl ester*: C<sub>21</sub>H<sub>21</sub>O<sub>2</sub>N. MW, 319. Yellow oil. B.p. 235-40°/2 mm.

*Allyl ester*: atquinol. Needles from EtOH. M.p. 30°. B.p. 265°/15 mm., 215°/0.8 mm. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Insol. H<sub>2</sub>O. *B,HCl*: yellow needles from EtOH. M.p. 145-7°. Insol. Et<sub>2</sub>O.

*Phenyl ester*: cryst. from EtOH. M.p. 132°.

*p-Chlorophenyl ester*: m.p. 117-18°.

*2-Naphthyl ester*: yellow cryst. M.p. 130°.

*Benzyl ester*: yellow cryst. from EtOH. M.p. 77-8°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Phenylethyl ester*: m.p. 72°.

*Cinnamyl ester*: cryst. from Et<sub>2</sub>O. M.p. 83°.

*Salicyl ester*: m.p. 188°.

*Chloride*: C<sub>16</sub>H<sub>10</sub>ONCl. MW, 267.5. Yellow cryst. from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 81-2°. *B,HCl*: m.p. about 153° decomp.

*Amide*: C<sub>16</sub>H<sub>12</sub>ON<sub>2</sub>. MW, 248. M.p. 196°.

*Nitrile*: C<sub>16</sub>H<sub>10</sub>N<sub>2</sub>. MW, 230. Needles from EtOH. M.p. 140°. B.p. about 365°.

*Anhydride*: C<sub>32</sub>H<sub>20</sub>O<sub>2</sub>N<sub>2</sub>. MW, 480. Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 185°. *Dimethiodide*: m.p. 132°.

*Anilide*: cryst. from EtOH. M.p. 198°.

*Hydrazide*: needles from EtOH. M.p. 222°. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Phenylhydrazide*: cryst. from AcOH. M.p. 215°.

*Azide*: needles from pet. ether. M.p. 87°.

*Methochloride*: prisms + 2H<sub>2</sub>O from H<sub>2</sub>O. M.p. 209-10° decomp., after turning green at about 140°.

*Methiodide*: red cryst. from EtOH or H<sub>2</sub>O. M.p. 160-5° decomp.

Du Puis, Lindwall, *J. Am. Chem. Soc.*, 1935, **56**, 471.

John, *Ber.*, 1926, **59**, 1447.

Rojahn, Schulten, *Arch. Pharm.*, 1926, **264**, 348.

Rosenmund, *Ber.*, 1921, **54**, 2893.

Wülfing, E.P., 325,985, (*Chem. Zentr.*, 1930, II, 93).

Boeringer Sohn, D.R.P., 485,426, (*Chem. Zentr.*, 1931, I, 853); D.R.P., 520,922, (*Chem. Zentr.*, 1931, I, 3173).

Gesellschaft für Chemische Industrie in Basel, U.S.P., 1,378,343, (*Chem. Zentr.*, 1921, IV, 514).

Bayer, D.R.P., 290,703, (*Chem. Zentr.*, 1916, I, 645).

Kalle, D.R.P., 287,304, (*Chem. - Zentr.*, 1915, II, 933).

**3-Phenylquinoline-4-carboxylic Acid**

(3-Phenylcinchoninic acid).

Prisms from AcOH. M.p. 273°. Somewhat difficultly sol. AcOH, Me<sub>2</sub>CO. At 290° → 3-phenylquinoline.

*Me ester*: needles from MeOH.Aq. M.p. 73°.

*Amide*: microcryst. powder from AcOH.Aq. M.p. 274°.

*Anilide*: needles from EtOH.Aq. M.p. 222°.

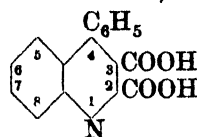
*Hydrazide*: platelets + 1H<sub>2</sub>O from EtOH.Aq. M.p. 154°.

Hübner, *Ber.*, 1906, **39**, 983.

**2-Phenylquinoline-6-carboxylic Acid.**

Cryst. from EtOH.Aq. M.p. 277°.

v. Braun, Brauns, *Ber.*, 1927, **60**, 1255.

**4-Phenylquinoline-2 : 3-dicarboxylic Acid (4-Phenylacridinic acid)**

C<sub>17</sub>H<sub>11</sub>O<sub>4</sub>N

MW, 293

M.p. 200-15°. Not obtained absolutely pure.

Claus, Nicolayson, *Ber.*, 1885, **18**, 2706.

## 2-Phenylquinoline-3 : 4-dicarboxylic Acid 454

### 2-Phenylquinoline - 3 : 4 - dicarboxylic Acid.

Needles + 2H<sub>2</sub>O from H<sub>2</sub>O. M.p. 193-4°. Mod. sol. EtOH. Spar. sol. H<sub>2</sub>O.

3-Nitrile : 2-phenyl-3-cyanocinchonic acid. C<sub>17</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>. MW, 274. Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 267-8°.

Engelhard, *J. prakt. Chem.*, 1898, **57**, 471.  
v. Meyer, *J. prakt. Chem.*, 1914, **90**, 23.

### 2-Phenylquinoline - 4 : 7 - dicarboxylic Acid.

Yellow cryst. powder. M.p. 200°. Sol. AcOEt. Spar. sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

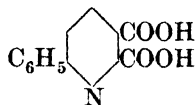
Neumann, D.R.P., 373,285, (*Chem. Zentr.*, 1923, IV, 665).

### 2-Phenylquinoline - 4 : 8 - dicarboxylic Acid.

Needles from EtOH or AcOH. Does not melt below 300°. Insol. H<sub>2</sub>O, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether. Heated with soda-lime → 2-phenylquinoline.

Doebner, Fettback, *Ann.*, 1894, **281**, 2.

### 6-Phenylquinolinic Acid (6-Phenylpyridine-2 : 3-dicarboxylic acid)



C<sub>13</sub>H<sub>9</sub>O<sub>4</sub>N MW, 243

M.p. 148-50° with decomp. to 6-phenylnicotinic acid.

Späth, Burger, *Monatsh.*, 1928, **49**, 270.

### Phenyl-2-quinolylamine.

See 2-Anilinoquinoline.

### Phenylquinolyethane.

See Phenylethylquinoline.

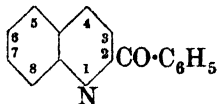
### Phenylquinolyethylene

See Styrylquinoline.

### sym.-Phenylquinolyhydrazine.

See under 2-Quinolyhydrazine.

### Phenyl 2-quinolyl Ketone (2-Benzoylquinoline)



C<sub>16</sub>H<sub>11</sub>ON MW, 233

Leaflets from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 111°.

Kaufmann, Dändikler, Burkhardt, *Ber.*, 1913, **48**, 2932.

Besthorn, *Ber.*, 1908, **41**, 2002.

## 2-Phenylquinoxaline

### Phenyl 4-quinolyl Ketone (4-Benzoylquinoline).

Needles from H<sub>2</sub>O. M.p. 60°. B.p. 220-4°/15 mm., 155°/0.5 mm. Sol. usual org. solvents except ligroin. Spar. sol. hot H<sub>2</sub>O.

B<sub>2</sub>HCl: plates from EtOH. Decomp. about 204°.

Oxime: amorphous. B<sub>2</sub>HCl: needles from MeOH. M.p. 256° decomp.

Phenylhydrazone: yellow cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 239-40°.

Picrate: yellow cryst. from EtOH. M.p. 214°.

Picrolonate: yellow cryst. from EtOH. Decomp. at 174°.

Methiodide: orange leaflets. M.p. 218° (not sharp).

Kaufmann, Peyer, *Ber.*, 1913, **48**, 60.

Rabe, Pasternack, *ibid.*, 1029.

Note.—Remfrey, Decker, *Ber.*, 1908, **41**, 1008, give m.p. of phenyl 4-quinolyl ketone as 294°.

### Phenyl 8-quinolyl Ketone (8-Benzoylquinoline).

Plates from MeOH. M.p. 94°. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin.

B<sub>2</sub>H<sub>2</sub>Pic: yellowish cryst. M.p. 213° decomp.

Oxime: two forms. (i) Cryst. + 1H<sub>2</sub>O from EtOH. M.p. 121°. (ii) cryst. + 1H<sub>2</sub>O from EtOH. M.p. 165°.

Phenylhydrazone: yellowish needles from EtOH. M.p. 190°.

Semicarbazone: cryst. from EtOH.Aq. M.p. 188°.

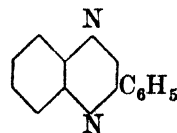
Azine: yellowish needles from C<sub>6</sub>H<sub>6</sub>. M.p. 287°.

Howitz, Köpke, *Ann.*, 1913, **396**, 42.

### Phenylquinolylmethane.

See Benzylquinoline.

### 2-Phenylquinoxaline



C<sub>14</sub>H<sub>10</sub>N<sub>2</sub> MW, 206

Needles from EtOH. M.p. 78°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, pet. ether. Sol. conc. H<sub>2</sub>SO<sub>4</sub> and conc. HCl with yellow col.

Hinsberg, *Ann.*, 1896, **292**, 246.

Fischer, Römer, *Ber.*, 1908, **41**, 2350.

**Phenyl-S Acid.**

See *N*-Phenyl-1-amino-8-naphthol-4-sulphonic Acid.

**Phenyl salicylate.**

See Salol.

**Phenylsalicylic Acid.**

See 3-Hydroxydiphenyl-2-carboxylic Acid and 2-Hydroxydiphenyl-3-carboxylic Acid.

**1-Phenylsarcosine.**

See  $\alpha$ -Methylaminophenylacetic Acid.

***N*-Phenylsarcosine** (*N*-Methylphenylglycine, *N*-methylanilinoacetic acid)

$\text{CH}_3$   
 $\text{C}_6\text{H}_5 \cdot \text{N} \cdot \text{CH}_2 \cdot \text{COOH}$

$\text{C}_9\text{H}_{11}\text{O}_2\text{N}$  MW, 165

Cryst. from  $\text{Et}_2\text{O}$ . M.p. 95–100°. Sol.  $\text{EtOH}$ . Mod. sol.  $\text{H}_2\text{O}$ .

*B.HCl*: prisms from conc.  $\text{HCl}$ . M.p. 210° (204°).

*Me ester*:  $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$ . MW, 179. B.p. 140–1°/10 mm. *Methiodide*: yellowish cryst. from  $\text{H}_2\text{O}$ . M.p. 98–9°.

*Et ester*:  $\text{C}_{11}\text{H}_{15}\text{O}_2\text{N}$ . MW, 193. B.p. 156–7°/17 mm., 148°/12 mm. *Methiodide*: cryst. from  $\text{H}_2\text{O}$ . Decomp. at 129°.

*Propyl ester*:  $\text{C}_{12}\text{H}_{17}\text{O}_2\text{N}$ . MW, 207. B.p. 175°/24 mm.

*Isoamyl ester*:  $\text{C}_{13}\text{H}_{19}\text{O}_2\text{N}$ . MW, 221. B.p. 300–2°, 179–81°/17 mm.

*l*-Menthyl ester: plates from  $\text{EtOH}$ . M.p. 83–5–84–5°.  $[\alpha]_D^{20}$  — 51–4° in  $\text{MeOH}$ .

*Amide*:  $\text{C}_9\text{H}_{12}\text{ON}_2$ . MW, 164. Prisms from hot  $\text{H}_2\text{O}$ . M.p. 163°. Mod. sol.  $\text{EtOH}$ .

*Nitrile*:  $\text{C}_9\text{H}_{10}\text{N}_2$ . MW, 146. M.p. 13°. B.p. 266°, 161–3°/21 mm., 148–9°/13 mm. *Methiodide*: cryst. powder from  $\text{EtOH}$ – $\text{Et}_2\text{O}$ . M.p. 100° decomp.

*Methochloride*: prisms from  $\text{H}_2\text{O}$ . M.p. 194–6° decomp.

*Methopicate*: needles from  $\text{EtOH}$ . M.p. 195°.

*Methochloroplatinate*: prisms from  $\text{EtOH}$ . M.p. 164° decomp.

Willstätter, Kahn, *Ber.*, 1904, 37, 416.

Gault, *Bull. soc. chim.*, 1908, 3, 373.

Knoevenagel, *Ber.*, 1904, 37, 4083.

Öchslein, *Ann. chim.*, 1914, 1, 243.

Warunis, Sachs, *Ber.*, 1904, 37, 2637.

**1-Phenylsemicarbazide**

$\text{H}_2\text{N} \cdot \text{CO} \cdot \text{NH} \cdot \text{NH} \cdot \text{C}_6\text{H}_5$

$\text{C}_7\text{H}_9\text{ON}_3$  MW, 151

Leaflets from  $\text{H}_2\text{O}$  or  $\text{EtOH}$ .Aq. M.p. 172°. Sol.  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ , hot  $\text{H}_2\text{O}$ . Spar. sol.

$\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin, cold  $\text{H}_2\text{O}$ . Fuming  $\text{HCl}$   $\rightarrow \text{CO}_2$ ,  $\text{NH}_3$  and phenylhydrazine.

*1-Nitroso*: yellow needles from  $\text{H}_2\text{O}$ . M.p. 126–7° decomp.

*4-Me*: m.p. 154–5°.

*4-Et*: plates from  $\text{EtOH}$ .Aq. M.p. 151°.

*1-Nitroso*: yellow needles from  $\text{Me}_2\text{CO}$ . M.p. 86–5° decomp.

*1-Acetyl*: see *unsym.*-Acetylphenylsemicarbazide.

Widman, *Ber.*, 1893, 26, 2613 (Footnote).

Fischer, *Ann.*, 1877, 190, 113.

Willstätter, Stoll, *Ber.*, 1909, 42, 4876.

**2-Phenylsemicarbazide**

$\text{C}_6\text{H}_5$   
 $\text{H}_2\text{N} \cdot \text{CO} \cdot \text{N} \cdot \text{NH}_2$

$\text{C}_7\text{H}_9\text{ON}_3$  MW, 151

Needles from  $\text{EtOH}$ . M.p. 120°. Sol.  $\text{EtOH}$ , warm  $\text{H}_2\text{O}$ . Very spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Reduces warm Fehling's.

*B<sub>2</sub>Cu(NO<sub>3</sub>)<sub>2</sub>*: blue cryst. from  $\text{H}_2\text{O}$ . M.p. 155°.

*Hydrochloride*: m.p. 185–6°.

*4-Et*:  $\text{C}_9\text{H}_{13}\text{ON}_3$ . MW, 179. Plates from  $\text{EtOH}$ .Aq. M.p. 88°.

*Picrate*: yellow needles from  $\text{EtOH}$ . M.p. 163°.

Busch, Walter, *Ber.*, 1903, 36, 1359.

Pellizzari, *Gazz. chim. ital.*, 1907, 37, i, 621.

**4-Phenylsemicarbazide**

$\text{C}_6\text{H}_5 \cdot \text{NH} \cdot \text{CO} \cdot \text{NH} \cdot \text{NH}_2$

$\text{C}_7\text{H}_9\text{ON}_3$  MW, 151

Plates from  $\text{H}_2\text{O}$ , needles from  $\text{C}_6\text{H}_6$ . M.p. 128°. Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ , dil. acids and alkalis. Spar. sol. hot  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ .

*B.HCl*: prisms. M.p. 215°. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*2-Me*:  $\text{C}_8\text{H}_{11}\text{ON}_3$ . MW, 165. M.p. 93–4°.

*1:1-Di-Me*:  $\text{C}_9\text{H}_{13}\text{ON}_3$ . MW, 179. M.p. 108°.

Wheeler, *Organic Syntheses*, Collective Vol. I, 439.

Sah, Ma, *J. Chinese Chem. Soc.*, 1934, 2, 32.

**2-Phenylserine.**

See 2-Hydroxy-1-amino-2-phenylpropionic Acid.

**8-Phenylstearic Acid**

$\text{C}_6\text{H}_5$   
 $\text{CH}_3 \cdot [\text{CH}_2]_8 \cdot \text{CH} \cdot [\text{CH}_2]_7 \cdot \text{COOH}$

$\text{C}_{24}\text{H}_{40}\text{O}_2$  MW, 360

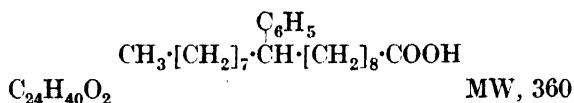


M.p. 36.5–38°. B.p. 200–4°/0.09 mm.  $D_{25}^{25}$  0.9340.  $n_D^{20}$  1.4891.

*p*-Bromophenacyl ester: cryst. from EtOH. M.p. 83.5–84.5°.

Harmon, Marvel, *J. Am. Chem. Soc.*, 1932, 54, 2515.

### 9-Phenylstearic Acid



M.p. 40–41.5°. B.p. 199–205°/0.09 mm.  $D_{25}^{25}$  0.9338.  $n_D^{20}$  1.4894.

*p*-Bromophenacyl ester: cryst. from EtOH. M.p. 71–2°.

See previous reference.

### $\alpha$ -Phenylstilbene.

See 1 : 1 : 2-Triphenylethylene.

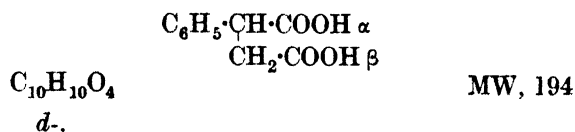
### Phenyl styryl Ketone.

See Chalkone.

### Phenylstyrylmethane.

See 1 : 3-Diphenylpropylene.

### Phenylsuccinic Acid



*d*-. Prisms from H<sub>2</sub>O. M.p. 173–4°. Sol. MeOH, EtOH, Me<sub>2</sub>CO.  $[\alpha]_D^{16.5} + 148.3^\circ$  in EtOH,  $[\alpha]_D^{18.4} + 173.4^\circ$  in Me<sub>2</sub>CO.

*Di-Me ester*: C<sub>12</sub>H<sub>14</sub>O<sub>4</sub>. MW, 222. B.p. 161–2°/16 mm.  $[\alpha]_D^{15} + 169.8^\circ$  in CCl<sub>4</sub>,  $[\alpha]_D^{19} + 140.9^\circ$  in EtOH. Racemised by CH<sub>3</sub>ONa in MeOH.

*Di-Et ester*: C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>. MW, 250. B.p. 166°/13 mm.  $[\alpha]_D^{13} + 103.4^\circ$  in Me<sub>2</sub>CO,  $[\alpha]_D^{15} + 130.6^\circ$  in CCl<sub>4</sub>,  $[\alpha]_D^{15.5} + 100.8^\circ$  in EtOH. Very rapidly racemised by CH<sub>3</sub>ONa.

*Anhydride*: C<sub>10</sub>H<sub>8</sub>O<sub>3</sub>. MW, 176. Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 83.5–84.5°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. CCl<sub>4</sub>, pet. ether.  $[\alpha]_D^{15} + 100.9^\circ$  in C<sub>6</sub>H<sub>6</sub>.

*l*-. M.p. 173–4°.  $[\alpha]_D^{14.5} - 173.3^\circ$  in Me<sub>2</sub>CO,  $[\alpha]_D^{13.5} - 147.1^\circ$  in MeOH. Not racemised by CH<sub>3</sub>ONa.

*Anhydride*: m.p. 83.5–84.5°.  $[\alpha]_D^{14} - 100.9^\circ$  in C<sub>6</sub>H<sub>6</sub>.

*dl*-. Leaflets from H<sub>2</sub>O. M.p. 168°. Very sol. EtOH, Et<sub>2</sub>O, AcOH, Me<sub>2</sub>CO. Sol. hot H<sub>2</sub>O.

Very spar. sol. CHCl<sub>3</sub>, CS<sub>2</sub>. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin, pet. ether.  $k = 1.64 \times 10^{-4}$  at 25°.

*$\alpha$ -Me ester*: C<sub>11</sub>H<sub>12</sub>O<sub>4</sub>. MW, 208. Cryst. from H<sub>2</sub>O. M.p. 92°. Sol. MeOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.  $k = 1.1 \times 10^{-4}$  at 25°.  *$\beta$ -Amide*: C<sub>11</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 207. Cryst. from MeOH.Aq. M.p. 119°.  *$\beta$ -Anilide*: needles from EtOH. M.p. 149°.  *$\beta$ -p-Toluidide*: m.p. 118°.

*$\beta$ -Me ester*: prisms from H<sub>2</sub>O. M.p. 102–3°. Sol. MeOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin, pet. ether.  $k = 0.49 \times 10^{-4}$  at 25°.  *$\alpha$ -Amide*: cryst. from H<sub>2</sub>O. M.p. 145°.  *$\alpha$ -Nitrile*: C<sub>11</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 189. Prisms from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 55°. B.p. 155–9°/10 mm.  *$\alpha$ -Anilide*: cryst. from MeOH.Aq. M.p. 96°.  *$\alpha$ -p-Toluidide*: m.p. 118°.

*Di-Me ester*: prisms from pet. ether. M.p. 57.5–58.5°. B.p. 160–2°/12 mm. Sol. MeOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*$\alpha$ -Et ester*: C<sub>12</sub>H<sub>14</sub>O<sub>4</sub>. MW, 222. Cryst. from MeOH. M.p. 88–9°.  *$\beta$ -Amide*: C<sub>12</sub>H<sub>15</sub>O<sub>3</sub>N. MW, 221. Cryst. from EtOH.Aq. M.p. 148–50°.

*$\beta$ -Et ester*: cryst. from MeOH. M.p. 95–6°.  *$\alpha$ -Amide*: cryst. from EtOH. M.p. 173°.  *$\alpha$ -Nitrile*: C<sub>12</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 203. Oil. B.p. 176°/16 mm.

*Di-Et ester*: liq. B.p. 160°/10 mm.

*Dichloride*: C<sub>10</sub>H<sub>8</sub>O<sub>2</sub>Cl<sub>2</sub>. MW, 231. Liq. B.p. 150–1°/12 mm.

*$\alpha$ -Amide*: C<sub>10</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 193. Plates from H<sub>2</sub>O. M.p. 158–9°. Sol. EtOH, Me<sub>2</sub>CO. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

*$\beta$ -Amide*: cryst. from H<sub>2</sub>O. M.p. 144–5°. Very spar. sol. C<sub>6</sub>H<sub>6</sub>.

*Diamide*: C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 192. M.p. 209–10°.

*$\alpha$ -Nitrile*: C<sub>10</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 175. Needles from EtOH.Aq. M.p. 150°. B.p. 215–18°/10 mm. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

*Dinitrile*: C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>. MW, 156. Cryst. from H<sub>2</sub>O. M.p. 68–9°. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>.

*Anhydride*: needles from Et<sub>2</sub>O. M.p. 54°. B.p. 204–6°/22 mm., 191–2°/12 mm.

*Imide*: C<sub>10</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 175. Prisms from AcOH.Aq. M.p. 90°. B.p. 205–10°/10 mm. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOH. Spar. sol. H<sub>2</sub>O. *N-Phenyl*: needles from AcOEt. M.p. 138°. *N-p-Tolyl*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 139°.

*$\alpha$ -Anilide*: m.p. 175°.

*$\beta$ -Anilide*: cryst. from MeOH. M.p. 170–1°.

*Dianilide*: m.p. 222°.

*$\alpha$ -p-Toluidide*: m.p. 175°.

*$\beta$ -p-Toluidide*: m.p. 168–9°.

**Phenylsulphamic Acid**

457

*Dihydrazide* : prisms from  $H_2O$ . M.p.  $174.5^\circ$ .  
*Dihydrochloride* : m.p.  $150^\circ$ .

Wren, Williams, *J. Chem. Soc.*, 1916, **109**, 574.

Lapworth, Baker, *Organic Syntheses*, Collective Vol. I, 440.

Curtius *et al.*, *J. prakt. Chem.*, 1930, **125**, 68.

Ramart-Lucas, Papadakis, *Ann. chim.*, 1932, **18**, 32.

**Phenylsulphamic Acid** (*Aniline- $\omega$ -sulphonic acid*)


 $C_6H_7O_3NS$ 

MW, 173

Plates from  $EtOH-Et_2O$ . Does not melt below  $280^\circ$ . Sol.  $H_2O$ . Decomp. by steam.

*NH<sub>4</sub> salt* : plates from  $EtOH.Aq$ . M.p.  $152^\circ$ . Sol.  $H_2O$ ,  $MeOH$ . Spar. sol.  $EtOH$ . Insol.  $Et_2O$ ,  $AcOEt$ ,  $C_6H_6$ , ligroin.

*Amide* :  $C_6H_8O_2N_2S$ . MW, 172. M.p.  $108.5-109^\circ$ .

*Dimethylamide* : needles. M.p.  $84-5^\circ$ .

*Anilide* : m.p.  $114^\circ$ .

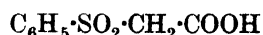
Traube, *Ber.*, 1890, **23**, 1654.

Paal, Kretzschmer, *Ber.*, 1894, **27**, 1244.

Bühner, *Ann.*, 1904, **333**, 288.

Battegay, Meybeck, *Compt. rend.*, 1932, **194**, 186.

**Phenylsulphoneacetic Acid** (*Methyl phenyl sulphone  $\omega$ -carboxylic acid*)


 $C_8H_8O_4S$ 

MW, 200

Needles from  $Et_2O$ . M.p.  $111.5-112.5^\circ$ . Sol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ . Mod. sol.  $C_6H_6$ . Decomp. at  $160^\circ \rightarrow CO_2 +$  methyl phenyl sulphone.

*Et ester* :  $C_{10}H_{12}O_4S$ . MW, 228. Prisms from  $EtOH$ . M.p.  $45^\circ$ . Sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

*Chloride* :  $C_8H_7O_3ClS$ . MW, 218.5. Cryst. from  $Et_2O$ . M.p.  $58^\circ$ .

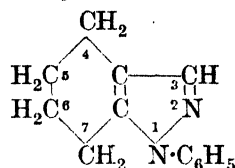
*Amide* :  $C_8H_9O_3NS$ . MW, 199. Needles from  $H_2O$ . M.p.  $156^\circ$ . Sol.  $H_2O$ ,  $EtOH$ . Spar. sol.  $Et_2O$ .

*Nitrile* :  $C_8H_7O_2NS$ . MW, 181. Needles. M.p.  $114^\circ$ . Sol.  $EtOH$ .

Troeger, Hille, *J. prakt. Chem.*, 1905, **71**, 225.

Otto, *J. prakt. Chem.*, 1884, **30**, 339.

Troeger, Vasterling, *J. prakt. Chem.*, 1905, **72**, 338.

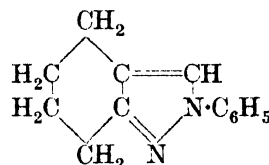
**1-Phenyl-1 : 2 : 3 : 4-tetrahydroisoquinoline****1-Phenyltetrahydroindazole**
 $C_{13}H_{14}N_2$ 

MW, 198

Plates from ligroin. M.p.  $58-9^\circ$ . B.p.  $178^\circ/10$  mm. Sol. ord. org. solvents.

*Picrate* : yellow cryst. from  $C_6H_6$ . M.p.  $125-6^\circ$ .

Auwers, Buschmann, Heidenreich, *Ann.*, 1924, **435**, 315.

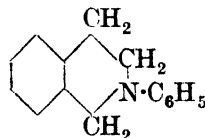
**2-Phenyltetrahydroindazole**
 $C_{13}H_{14}N_2$ 

MW, 198

Prisms. M.p.  $48.5-49.5^\circ$ . B.p.  $177^\circ/10$  mm. Sol. ord. org. solvents.

*Picrate* : plates from  $EtOH$ . M.p.  $126.5-127.5^\circ$ .

See previous reference.

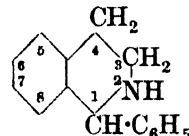
**N - Phenyl - 1 : 2 : 3 : 4 - tetrahydroisoquinoline**
 $C_{15}H_{15}N$ 

MW, 209

B.p.  $198^\circ/16$  mm. Turns brown rapidly in air.

*Picrate* : yellow plates from  $EtOH$ . M.p.  $120^\circ$ .

v. Braun, Zobel, *Ber.*, 1923, **56**, 2152.

**1 - Phenyl - 1 : 2 : 3 : 4 - tetrahydroisoquinoline**
 $C_{15}H_{15}N$ 

MW, 209

Cryst. from pet. ether. M.p.  $84^\circ$ .  $[\alpha]_D^{20} - 12.4^\circ$  in  $CHCl_3$ .

*B.HCl* : cryst. from  $EtOH-Et_2O$ . M.p.  $204^\circ$ .

### 3-Phenyl-1 : 2 : 3 : 4-tetrahydroisoquinoline

458

*dl.*

Prisms from Et<sub>2</sub>O. M.p. 97°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, Et<sub>2</sub>O.

N-Me : C<sub>10</sub>H<sub>13</sub>N. MW, 147. Needles + 1H<sub>2</sub>O from EtOH.Aq. M.p. 120-30°. *Methiodide* : prisms from EtOH.Aq. M.p. 240-3°.

*Bitartrate* : cryst. from H<sub>2</sub>O. M.p. 187°.

Leithe, *Monatsh.*, 1929, 53 and 54, 961.

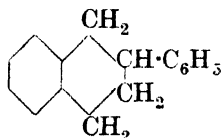
Freund, Bode, *Ber.*, 1909, 42, 1761.

### 3-Phenyl-1 : 2 : 3 : 4-tetrahydroisoquinoline.

Cryst. M.p. 45-8°. Readily sol. ord. org. solvents.

Gabriel, *Ber.*, 1885, 18, 3479.

### 2-Phenyl-1 : 2 : 3 : 4-tetrahydronaphthalene



C<sub>16</sub>H<sub>16</sub>

MW, 208

B.p. 180-1°/13 mm. D<sub>4</sub><sup>19</sup> 1.0579. n<sub>D</sub><sup>19</sup> 1.5980.

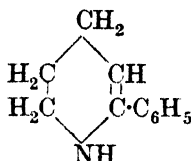
v. Braun, Manz, *Ann.*, 1929, 468, 267.

Späth, *Monatsh.*, 1912, 33, 1046.

### 1-Phenyl-1 : 2 : 3 : 4-tetrahydronaphthalene-1 : 4-dicarboxylic Acid.

See Isatropic Acid.

### 2-Phenyl-1 : 4 : 5 : 6-tetrahydropyridine



C<sub>11</sub>H<sub>13</sub>N

MW, 159

F.p. about 18°. B.p. 275-7°/751 mm.

B,HCl : cryst. from HCl. M.p. 86-7°.

B,HAuCl<sub>4</sub> : cryst. M.p. 118°.

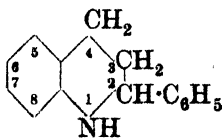
B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub> : orange-red plates. M.p. 210-11° decomp.

*Picrate* : yellow needles. M.p. 181°.

Gabriel, *Ber.*, 1908, 41, 2012.

Cloke, Ayers, *J. Am. Chem. Soc.*, 1934, 56, 2145.

### 2-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline



C<sub>15</sub>H<sub>15</sub>N

MW, 209

### 6-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline

Viscous oil. B.p. 341-4°, 196-7°/12 mm. Sol. Et<sub>2</sub>O, hot EtOH. Insol. H<sub>2</sub>O.

N-Me : C<sub>16</sub>H<sub>17</sub>N. MW, 223. Needles from ligroin. M.p. 106-7°.

Doebner, Miller, *Ber.*, 1886, 19, 1198.

Freund, *Ber.*, 1904, 37, 4669.

v. Braun, Petzold, Seemann, *Ber.*, 1922, 55, 3785.

### 3-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Cryst. from EtOH. M.p. 83°.

B,HCl : needles from EtOH.Aq. M.p. 229°.

N-Acetyl : prisms from EtOH.Aq. M.p. 78°.

N-Nitroso : m.p. 147°.

*Picrate* : cryst. from EtOH. M.p. 181°.

*Picrolonate* : yellow powder from EtOH. M.p. 205°.

v. Braun, Petzold, Seemann, *Ber.*, 1922, 55, 3790.

### 4-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Plates from EtOH.Aq. M.p. 74°.

B,HCl,1H<sub>2</sub>O : needles. M.p. 193-4°. Sublimes at 140°.

*Sulphate* : needles. M.p. 158°. Spar. sol. H<sub>2</sub>O.

B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub> : m.p. 215° decomp.

N-Me : oil. *Picrate* : yellow needles from EtOH. M.p. 222-4°.

N-Acetyl : plates from EtOH. M.p. 120°.

N-Benzoyl : needles from Et<sub>2</sub>O. M.p. 147°.

N-Nitroso : yellow needles from EtOH. M.p. 72°.

*Picrate* : yellow needles from EtOH. M.p. 183°.

Koenigs, Meimberg, *Ber.*, 1895, 28, 1042.

Höchst, D.R.P., 79,385.

### 6-Phenyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Amorphous solid. Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

B,HCl,1½H<sub>2</sub>O : needles. M.p. 204°. Spar. sol. H<sub>2</sub>O.

N-Me : amorphous powder from Et<sub>2</sub>O. Sol. hot H<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>. Insol. CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>.

*Methiodide* : prisms from EtOH. M.p. 194-5°. Insol. Et<sub>2</sub>O. *Picrate* : yellow needles from EtOH. M.p. 147°.

N-Acetyl : needles from EtOH. M.p. 99-100°.

N-Benzoyl : needles from EtOH.Aq. M.p. 137°.

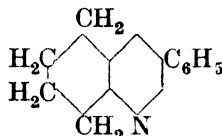
N-Nitroso : yellow cryst. from pet. ether. M.p. 111-12°.

### 3-Phenyl-5 : 6 : 7 : 8-tetrahydroquinoline

*Picrate*: yellow needles from  $H_2O$ . M.p.  $165^\circ$ .

La Coste, *Ann.*, 1885, **230**, 20.

### 3-Phenyl-5 : 6 : 7 : 8-tetrahydroquinoline



$C_{15}H_{15}N$  MW, 209

Colourless oil. B.p.  $211-12^\circ$ . Misc. with EtOH in all proportions.

*B, HCl*: m.p.  $235^\circ$ .

*Picrolonate*: yellow powder from EtOH. M.p.  $201^\circ$ .

*Methiodide*: m.p.  $240-3^\circ$ .

v. Braun, Petzold, Seemann, *Ber.*, 1922, **55**, 3791.

### 1-Phenyltetramethylene Glycol (1-Phenyl-1 : 4-butandiol, $\alpha\delta$ -dihydroxybutylbenzene)

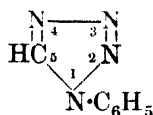


$C_{10}H_{14}O_2$  MW, 166

Cryst. from  $Et_2O$ . M.p.  $75^\circ$ . Readily sol. org. solvents.

Marshall, Perkin, *J. Chem. Soc.*, 1891, **59**, 890.

### 1-Phenyl-1 : 2 : 3 : 4-tetrazole



$C_7H_6N_4$  MW, 146

Cryst. from EtOH.Aq. M.p.  $65-6^\circ$ . Sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ .

*AgNO\_3 add. comp.*: needles. Decomp. at  $126^\circ$ .

*HgCl\_2 add. comp.*: needles. M.p.  $147^\circ$ .

Stollé, Henke-Stark, *J. prakt. Chem.*, 1930, **124**, 281.

Freund, Paradies, *Ber.*, 1901, **34**, 3120.

Dimroth, de Montmollin, *Ber.*, 1910, **43**, 2907.

### 5-Phenyl-1 : 2 : 3 : 4-tetrazole.

Needles from EtOH. M.p.  $215^\circ$  decomp.

*Mercuriacetate*: needles from EtOH. M.p.  $140^\circ$  decomp.

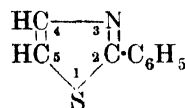
Knoll, D.R.P., 521,870, (*Chem. Abstracts*, 1931, **25**, 3364).

Stollé, Henke-Stark, *J. prakt. Chem.*, 1930, **124**, 287.

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### Phenyl 2-thienyl Ketone

#### 2-Phenylthiazole



$C_9H_7NS$  MW, 161

B.p.  $267-9^\circ/732$  mm. ( $263-4^\circ/772$  mm.).

*B, HCl*: needles +  $2H_2O$ . M.p.  $61-2^\circ$ .

*B\_2, H\_2PtCl\_6*: yellow micro-cryst. +  $2H_2O$ . M.p.  $198-9^\circ$  ( $173-5^\circ$ ) decomp.

*Picrate*: m.p.  $124-5^\circ$ .

Hubacher, *Ann.*, 1890, **259**, 234.

Bachstetz, *Ber.*, 1914, **47**, 3164.

#### 4-Phenylthiazole.

M.p.  $52^\circ$ . B.p.  $273^\circ$ . Sol. EtOH.

*B, H\_2AuCl\_4*: m.p.  $174-5^\circ$  decomp.

*B\_2, H\_2PtCl\_6*: brownish-yellow needles +  $2H_2O$ . M.p.  $196^\circ$ .

*Picrate*: m.p.  $164-5^\circ$ .

Popp, *Ann.*, 1889, **250**, 279.

#### 5-Phenylthiazole.

M.p.  $45-6^\circ$ .

*B, H\_2AuCl\_4*: m.p.  $185^\circ$ .

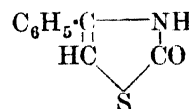
*B\_2, H\_2PtCl\_6*: yellow micro-cryst. Decomp. at  $281-2^\circ$ .

*B\_2, H\_2Cr\_2O\_7*: needles from HCl. M.p.  $108-9^\circ$  decomp.

*Picrate*: yellow needles from EtOH. M.p.  $138-9^\circ$  decomp.

Bachstetz, *Ber.*, 1914, **47**, 3164.

#### 4-Phenylthiazolone



$C_9H_7ONS$  MW, 177

M.p.  $204^\circ$ . Spar. sol. org. solvents. Sol. alkalis.

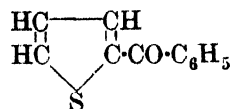
*Hydrazone*: needles from EtOH. M.p.  $167-8^\circ$ . *N-Acetyl*: needles from EtOH. M.p.  $196-7^\circ$ .

Dyckerhoff, *Ber.*, 1877, **10**, 120.

Arapides, *Ann.*, 1888, **249**, 15.

Bose, *Quart. J. Indian Chem. Soc.*, 1924, **I**, 60.

### Phenyl 2-thienyl Ketone (2-Benzoylthiophene)



$C_{11}H_8OS$

MW, 188

## Phenylthioacetic Acid

Needles from pet. ether. M.p. 55–6°. B.p. 300°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Oxime*:  $\alpha$ -form, needles from EtOH. M.p. 93°. *Acetyl*: m.p. 80°.  $\beta$ -Form: m.p. 113–14°. *Acetyl*: m.p. 88–9°.

Minnis, *Organic Syntheses*, 1932, XII, 62.  
Steinkopf, D.R.P., 297,203, (*Chem. Zentr.*, 1917, I, 834).

Hantzsch, *Ber.*, 1891, 24, 60.

### Phenylthioacetic Acid (Thio- $\alpha$ -toluic acid)

$C_6H_5 \cdot CH_2 \cdot CO \cdot SH$  or  $C_6H_5 \cdot CH_2 \cdot CSOH$   
 $C_8H_8OS$  MW, 152

Oil. Standing in air slowly  $\rightarrow$  di-phenylacetyl disulphide, m.p. 62°.

*Et ester*:  $C_{10}H_{12}OS$ . MW, 180. B.p. 170°/99 mm.  $D_4^{25}$  1.0142.

*Amide*: phenylthioacetamide.  $C_8H_9NS$ . MW, 151. Cryst. from EtOH. M.p. 97.5–98°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. hot H<sub>2</sub>O. Dist.  $\rightarrow$  benzyl cyanide.

Johnson, *J. Am. Chem. Soc.*, 1906, 28, 1457.

Bernthsen, *Ann.*, 1877, 184, 293.

### Phenylthiocarbamic Acid (Thiocarbamic acid)

$C_6H_5 \cdot NH \cdot CSOH$  or  $C_6H_5 \cdot NH \cdot COSH$   
 $C_7H_7ONS$  MW, 153

*O-Me ester*:  $C_8H_9ONS$ . MW, 167. Cryst. from EtOH. M.p. 97° (93–4°). Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*S-Me ester*: leaflets from EtOH.Aq. M.p. 83–4°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. Sol. conc. H<sub>2</sub>SO<sub>4</sub>, reppd. by H<sub>2</sub>O.

*Et ester*: see Phenylthiourethane.

*O-Propyl ester*:  $C_{10}H_{13}ONS$ . MW, 195. Needles from EtOH. M.p. 48°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

*O-Isopropyl ester*: cryst. from EtOH. M.p. 85.5°.

*O-Butyl ester*:  $C_{11}H_{15}ONS$ . MW, 209. Needles from Et<sub>2</sub>O–pet. ether. M.p. 53°.

*O-Isobutyl ester*: prisms. M.p. 80.5°.

*O-Isoamyl ester*:  $C_{12}H_{17}ONS$ . MW, 233. Cryst. M.p. about 21°.

*S-Isoamyl ester*: needles from Et<sub>2</sub>O. M.p. 67°.

*O-Allyl ester*: needles from ligroin. M.p. 64.5–65.5°.

*O-l-Menthyl ester*: cryst. from EtOH.Aq. M.p. 74–5°.  $[\alpha]_D^{20}$  – 63.07° in EtOH.

*O-Phenyl ester*:  $C_{13}H_{11}ONS$ . MW, 239. Needles from CHCl<sub>3</sub>. M.p. 142°. *Ag salt*:

## 460 Phenylthioglycollic Acid *o*-carboxylic Acid

bronze-yellow cryst. from CHCl<sub>3</sub>–EtOH. M.p. 186°.

*S-Phenyl ester*: needles from EtOH. M.p. 122–122.5°.

*O-Benzyl ester*: cryst. from EtOH. M.p. 82–82.5°.

*S-Benzyl ester*: prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 96–7°.

*Amide*: see Phenylthiourea.

Bettschart, Bistrzycki, *Helv. Chim. Acta*, 1919, 2, 131.

Schneider, Wrede, *Ber.*, 1914, 47, 2040.

Wheeler, Barnes, *Am. Chem. J.*, 1900, 24, 71.

Orndorff, Richmond, *Am. Chem. J.*, 1899, 22, 456.

### Phenylthioglycollic Acid (Phenylmercaptoacetic acid, thioanisole- $\omega$ -carboxylic acid, carboxymethyl phenyl sulphide)

$C_6H_5 \cdot S \cdot CH_2 \cdot COOH$   
 $C_8H_8O_2S$  MW, 168

Needles. M.p. 61–2°. Spar. sol. cold H<sub>2</sub>O.

*Me ester*:  $C_9H_{10}O_2S$ . MW, 182. B.p. 262–3°.  $D_{25}^{25}$  1.1728.  $n_D^{25}$  1.5569.

*Et ester*:  $C_{10}H_{12}O_2S$ . MW, 196. B.p. 265°, 144.5°/14 mm.  $D_{25}^{25}$  1.1322.  $n_D^{25}$  1.5429.

*Propyl ester*:  $C_{11}H_{14}O_2S$ . MW, 210. B.p. 270° decomp.  $D_{25}^{25}$  1.1021.  $n_D^{25}$  1.5348.

*Butyl ester*:  $C_{12}H_{16}O_2S$ . MW, 224. B.p. 280° decomp.  $D_{25}^{25}$  1.0816.  $n_D^{25}$  1.5308.

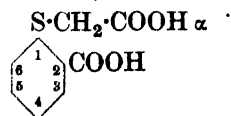
*Chloride*:  $C_8H_7OCIS$ . MW, 186.5. B.p. 115–16°/3 mm.  $D_{25}^{25}$  1.2581.

*Amide*:  $C_8H_9ONS$ . MW, 167. Needles from EtOH. M.p. 104°. Sol. EtOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O.

Uyeda, *J. Chem. Soc. Japan*, 1931, 410.

Pummerer, *Ber.*, 1910, 43, 1407.

### Phenylthioglycollic Acid *o*-carboxylic Acid (S-Carboxymethylthiosalicylic acid, *o*-carboxyphenylthioglycollic acid)



$C_9H_8O_4S$  MW, 212

Needles from AcOEt. M.p. 213°. Sol. EtOH, Me<sub>2</sub>CO, AcOH, AcOEt. Mod. sol. hot H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin.

*2-Me-ester*:  $C_{10}H_{10}O_4S$ . MW, 226. Cryst. from hot H<sub>2</sub>O. M.p. 151°.

$\alpha$ -*Me ester*: cryst. M.p. 126–127.5°.

*Di-Me ester*:  $C_{11}H_{12}O_4S$ . MW, 240. Needles from MeOH. M.p. 52°. Insol. H<sub>2</sub>O.

2-*Et ester*:  $C_{11}H_{12}O_4S$ . MW, 240. M.p. 137°.

2-*Nitrile*: *o*-cyanophenylthioglycollic acid.  $C_9H_7O_2NS$ . MW, 193. Yellow needles from  $H_2O$ . M.p. 140°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold  $H_2O$ ,  $C_6H_6$ , ligroin. *Me ester*:  $C_{10}H_8O_2NS$ . MW, 207. Needles from MeOH. M.p. 87-8°.

Friedländer, *Ann.*, 1907, **351**, 402.

Höchst, D.R.P., 216,725, (*Chem. Zentr.*, 1910, I, 130).

Lesser, D.R.P., 229,067, (*Chem. Zentr.*, 1911, I, 104).

Wegscheider, Joachimowitz, *Monatsh.*, 1914, **35**, 1049.

**Phenylthioglycollic Acid *p*-carboxylic Acid** (*p*-Carboxyphenylthioglycollic acid).

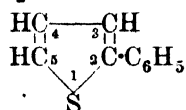
Yellow cryst. M.p. 267-9° decomp. Sol. EtOH, Me<sub>2</sub>CO, AcOH. Spar. sol.  $H_2O$ , Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ , ligroin.

*Di-Me ester*: needles from ligroin. M.p. 63-4°.

*Di-Et ester*:  $C_{13}H_{16}O_4S$ . MW, 268. Needles from ligroin. M.p. 98°.

Friedländer, Chwala, *Monatsh.*, 1907, **28**, 279.

**2-Phenylthiophene**



$C_{10}H_8S$  MW, 160

Needles from EtOH. M.p. 40-1°. Sol. EtOH, AcOEt. Mod. sol. Et<sub>2</sub>O,  $C_6H_6$ ,  $CS_2$ , ligroin. Volatile in steam.

Kues, Paal, *Ber.*, 1886, **19**, 3142.

**3-Phenylthiophene.**

Cryst. from EtOH. M.p. 91-4-92°. B.p. 254-60°/725 mm. Sol. ord. org. solvents. Volatile in steam.

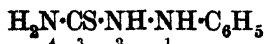
Bamberger, *Ber.*, 1897, **30**, 367.

Chrzaszczewska, *Chem. Zentr.*, 1926, II, 2905.

**Phenylthiosalicylic Acid.**

See Diphenyl sulphide 2-carboxylic Acid.

**1-Phenylthiosemicarbazide**



$C_7H_9N_3S$  MW, 167

Prisms from EtOH. M.p. 200-1° decomp. Mod. sol. hot EtOH. Spar. sol.  $H_2O$ , Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ .

4-*N-Me*: needles. M.p. 170° (163-4°).

4-*N-Acetyl*: prisms from EtOH. M.p. 178-9°.

4-*N-Propionyl*: prisms from EtOH. M.p. 155-6° decomp.

1-*N-Benzoyl*: cryst. from EtOH. M.p. 223°

4-*N-Benzoyl*: prisms from EtOH. M.p. 136-7°.

1:4-*N-Dibenzoyl*: needles. M.p. 188°.

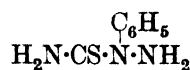
Fromm, Trnka, *Ann.*, 1925, **442**, 154.

Fischer, Besthorn, *Ann.*, 1882, **212**, 324.

Busch, Opfermann, Walther, *Ber.*, 1904, **37**, 2332.

Dixon, *J. Chem. Soc.*, 1889, **55**, 303; 1896, **69**, 860.

**2-Phenylthiosemicarbazide**



$C_7H_9N_3S$  MW, 167

Cryst. from hot  $H_2O$ . M.p. 153°. Sol. EtOH, hot  $H_2O$ . Spar. sol.  $C_6H_6$ .

4-*N-Me*: needles from EtOH.Aq. M.p. 91°. Sol. EtOH, Me<sub>2</sub>CO, AcOH. Mod. sol. Et<sub>2</sub>O,  $CHCl_3$ .

4-*N-Et*: prisms from EtOH. M.p. 121-2°.

*Picrate*: yellow cryst. from EtOH. M.p. 145°.

Pellizzari, *Gazz. chim. ital.*, 1907, **37**, 622.

Rolla, *Gazz. chim. ital.*, 1908, **38**, 345.

Dixon, *J. Chem. Soc.*, 1889, **55**, 302.

**4-Phenylthiosemicarbazide**



$C_7H_9N_3S$  MW, 167

Plates from EtOH. M.p. 140° decomp. Spar. sol.  $C_6H_6$ . Insol. ligroin.

2-*N-Me*: plates from EtOH. M.p. 143°.

1:2-*N-Di-Me*: prisms from EtOH. M.p. 115°.

2-*N-Et*: needles. M.p. 109-10°.

1:2-*N-Dibenzoyl*: yellow cryst. from EtOH. M.p. 195°.

1:1:2:4-*N-Tetrabenzoyl*: yellowish-green cryst. from EtOH. M.p. 148°.

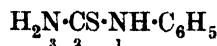
Fromm, Trnka, *Ann.*, 1925, **442**, 154.

Busch, *Ber.*, 1909, **42**, 4600.

Freund, Hempel, *Ber.*, 1895, **28**, 77.

Knorr, Köhler, *Ber.*, 1906, **39**, 3264.

**Phenylthiourea**



$C_7H_9N_2S$  MW, 152

Needles from hot  $H_2O$ . M.p. 154°. Sol. EtOH.

3-*N-Me*:  $C_8H_{10}N_2S$ . MW, 166. Needles. M.p. 113°. Sol. EtOH.

3-*N-Di-Me*:  $C_9H_{12}N_2S$ . MW, 180. Prisms from EtOH. M.p. 134–5°.

3-*N-Et*:  $C_9H_{12}N_2S$ . MW, 180. Cryst. from EtOH or  $C_6H_6$ . M.p. 99–99.5°.

3-*N-Di-Et*:  $C_{11}H_{16}N_2S$ . MW, 208. Yellow oil. B.p. 182°/15 mm.

1-*N-Acetyl*: prisms from EtOH.Aq. M.p. 139° decomp.

3-*N-Acetyl*: m.p. 173° (171°). Cryst. from dil. EtOH. Mod. sol. EtOH, Et<sub>2</sub>O. Prac. insol. H<sub>2</sub>O. Sol. dil. alkalis.

3-*N-Propionyl*: prisms from EtOH. M.p. 129–30°.

1-*N-Benzoyl*: cryst. from EtOH. M.p. about 140°.

3-*N-Benzoyl*: needles. M.p. 148–9°.

1-*N-Phenylacetyl*: prisms from EtOH. M.p. 113–14°.

Salkowski, *Ber.*, 1891, **24**, 2728.

Gebhardt, *Ber.*, 1884, **17**, 3038.

Streiger, *Monatsh.*, 1916, **37**, 649.

Schiff, *Ann.*, 1868, **148**, 338.

Hugershoff, *Ber.*, 1899, **32**, 3659.

### Phenylthiourethane

$C_6H_5 \cdot NH \cdot CS \cdot OC_2H_5$  (i)

$C_6H_5 \cdot NH \cdot CO \cdot SC_2H_5$  (ii)

$C_9H_{11}ONS$  MW, 181

(i) "Xanthogenanilide." Cryst. from EtOH. M.p. 71–2°. Insol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Sol. alkalis. Heat with EtI  $\rightarrow$  S-ether.

(ii) Needles from EtOH. M.p. 73°. Sol. EtOH. Insol. H<sub>2</sub>O.

Will, *Ber.*, 1882, **15**, 341.

Hofmann, *Ber.*, 1870, **3**, 774.

Jacobson, Klein, *Ber.*, 1893, **26**, 2364.

Biilmann, *Ann.*, 1906, **348**, 141.

### Phenyltoluene.

See Methylbiphenyl.

### 6-Phenyl-*m*-toluic Acid.

See 2-Methyldiphenyl-4-carboxylic Acid.

### *N*-Phenyltoluidine.

See Methyldiphenylamine.

### Phenyl-tolyl.

See Methylbiphenyl.

### Phenyltolylamine.

See Methyldiphenylamine.

### Phenyltolylcarbinol.

See Methylbenzhydrol.

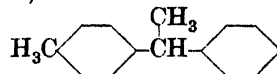
### Phenyl *p*-tolyl Diketone.

See 4-Methylbenzil.

### 4-*N*-Phenyl-1 : 3 : 4-tolylenediamine.

See 2-Amino-4-methyldiphenylamine.

### 1-Phenyl-1-*p*-tolylethane ( $\alpha$ -Methylphenyl-*p*-tolylmethane)



$C_{16}H_{18}$  MW, 196

Oil. B.p. 280–2°, 154–155.8°/14 mm.  $D_4^{17.2}$  0.9847.  $n_D^{17.2}$  1.56590.

Auwers, *Ber.*, 1916, **49**, 2401.

Kraemer, Spilker, *Ber.*, 1891, **24**, 2788.

### 1-Phenyl-2-*p*-tolylethane.

See 4-Methyldibenzyl.

### Phenyl tolyl Ether.

See under Cresol.

### 1-Phenyl-2-tolylethylene.

See Methylstilbene.

### Phenyl-*o*-tolylguanidine



$C_{14}H_{15}N_3$  MW, 225

Needles from AcOH. M.p. 123–5°. Sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. H<sub>2</sub>O.

Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 384.

Heuser, U.S.P., 1,669,242, (*Chem. Abstracts*, 1928, **22**, 2172).

### Phenyl-*p*-tolylguanidine.

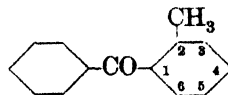
Needles from ligroin. M.p. 120–2°. Sol. EtOH,  $C_6H_6$ . Mod. sol. H<sub>2</sub>O.

Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 385.

### *sym.*-Phenyltolylhydrazine.

See Methylhydrazobenzene.

### Phenyl *o*-tolyl Ketone (2-Methylbenzophenone)



$C_{14}H_{12}O$  MW, 196

B.p. 309.5°/762 mm., 168°/12 mm.

*Oxime*: phenyl-*syn*-form. M.p. 105°. Phenyl-*anti*-form: m.p. 69°.

*Ketimide*: oil. B.p. 136–7°/4 mm.  $D_4^{18.5}$  1.0614.  $n_D^{18.5}$  1.6065.

Smith, *Ber.*, 1891, **24**, 4047.

Goldschmidt, Stöcker, *Ber.*, 1891, **24**, 2805.

### Phenyl *m*-tolyl Ketone (3-Methylbenzophenone).

Oil. B.p. 311–13°/723 mm.  $D_4^{17.5}$  1.088. Misc. with EtOH, Et<sub>2</sub>O,  $CHCl_3$ , AcOH,  $C_6H_6$ .

*Oxime*: cryst. from EtOH. M.p. 100–1°.

Ador, Rilliet, *Ber.*, 1879, **12**, 2300.

Goldschmidt, Stöcker, *Ber.*, 1891, **24**, 2807.

**Phenyl *p*-tolyl Ketone** (4-Methylbenzophenone).

Dimorphous. Stable form, m.p. 59–60°. Metastable form, m.p. 55°. B.p. 311–12°/720 mm. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH.

*Oxime*: two forms. (i) Needles from EtOH.Aq. M.p. 153–4°. *O-Me ether*: needles from EtOH.Aq. M.p. 70·5–72°. *N-Me*: plates from ligroin. M.p. 91–2°. *N-Acetyl*: prisms. M.p. 124–5°. (ii) Needles from EtOH. M.p. 136–137·5°. *O-Me ether*: oil. B.p. 184–5°/16 mm. *N-Me*: prisms from ligroin. M.p. 113–14°.

*Semicarbazone*: cryst. from EtOH. M.p. 121–2°.

*Hydrazone*: cryst. from EtOH. M.p. 80–1°.

2:4-Dinitrophenylhydrazone: m.p. 199–200°.

*Ketimide*: m.p. 37°. B.p. 147°/5 mm. D<sub>4</sub><sup>20</sup> 1·0617. n<sub>D</sub><sup>20</sup> 1·6097.

Staudinger, Goldstein, *Ber.*, 1916, **49**, 1926.

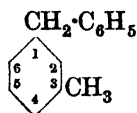
Marshall, *J. Chem. Soc.*, 1915, **107**, 516.

Meyer, *Monatsh.*, 1907, **28**, 1223.

Semper, Lichtenstadt, *Ber.*, 1918, **51**, 936.

Bruzau, *Ann. chim.*, 1934, **1**, 352.

**Phenyl-*m*-tolylmethane** (*m*-Benzyltoluene, 3-methyldiphenylmethane)



C<sub>14</sub>H<sub>14</sub> MW, 182

B.p. 275°/747 mm., 268–269·5°/725 mm. D<sub>17·5</sub><sup>17·5</sup> 0·997.

Ador, Rilliet, *Ber.*, 1879, **12**, 2300.

**Phenyl-*p*-tolylmethane** (*p*-Benzyltoluene, 4-methyldiphenylmethane).

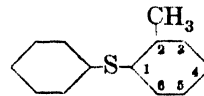
B.p. 279–80°, 145·5–155·7°/13·5 mm. D<sub>18</sub><sup>18</sup> 0·994. n<sub>D</sub><sup>14·7</sup> 1·56922. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH.

Weiler, *Ber.*, 1900, **33**, 464.

Hirst, Cohen, *J. Chem. Soc.*, 1895, **67**, 828.

v. Meyer, *J. prakt. Chem.*, 1910, **82**, 539.

**Phenyl *o*-tolyl sulphide** (2-Methyldiphenyl sulphide)



C<sub>13</sub>H<sub>12</sub>S MW, 200

Oil. B.p. 304·5°/724 mm., 222·5°/100 mm. D<sub>4</sub><sup>1</sup> 1·1131, D<sub>20</sub><sup>20</sup> 1·0893.

Bourgeois, *Ber.*, 1895, **28**, 2322.

Ziegler, *Ber.*, 1890, **23**, 2471.

**Phenyl *m*-tolyl sulphide** (3-Methyldiphenyl sulphide).

Oil. F.p. – 6·5°. B.p. 309·5°, 164·5°/11 mm. D<sub>4</sub><sup>1</sup> 1·1058, D<sub>15</sub><sup>15</sup> 1·0937.

Bourgeois, *Ber.*, 1895, **28**, 2323.

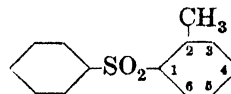
**Phenyl *p*-tolyl sulphide** (4-Methyldiphenyl sulphide).

Oil. F.p. 15·7°. B.p. 311·5°, 167·5°/11 mm. D<sub>10·7</sub><sup>10·7</sup> 1·0900.

See previous reference and also

Ziegler, *Ber.*, 1890, **23**, 2471.

**Phenyl *o*-tolyl sulphone** (2-Methyldiphenyl sulphone)



C<sub>13</sub>H<sub>12</sub>O<sub>2</sub>S MW, 232

Plates from EtOH. M.p. 80–1°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. Insol. H<sub>2</sub>O.

Ullmann, Lehner, *Ber.*, 1905, **38**, 734.

**Phenyl *m*-tolyl sulphone** (3-Methyldiphenyl sulphone).

Needles. M.p. 109°.

Courtot, Frenkiel, *Compt. rend.*, 1934, **199**, 558.

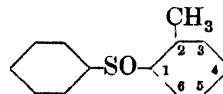
**Phenyl *p*-tolyl sulphone** (4-Methyldiphenyl sulphone).

Plates from EtOH. M.p. 124·5°. Spar. sol. C<sub>6</sub>H<sub>6</sub>, AcOH.

Michael, Adair, *Ber.*, 1878, **11**, 116.

Olivier, *Rec. trav. chim.*, 1914, **33**, 249.

**Phenyl *o*-tolyl sulphoxide**



C<sub>13</sub>H<sub>12</sub>OS MW, 216

Prisms. M.p. 42°. B.p. 220°/11 mm.

Courtot, Frenkiel, *Compt. rend.*, 1934, **199**, 558.



**Phenyl *m*-tolyl sulphoxide.**

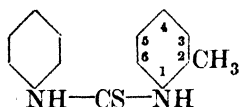
B.p. 215°/12 mm.

See previous reference.

**Phenyl *p*-tolyl sulphoxide.**

M.p. 73°.

See previous reference.

***N*-Phenyl-*N'*-*o*-tolylthiourea (o-Methylthiocarbanilide)** $C_{14}H_{14}N_2S$ 

MW, 242

Needles. M.p. 141–2°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Phenylhydrazine → 1-phenyl-4-*o*-tolylthiosemicarbazide.

Walther, Stenz, *J. prakt. Chem.*, 1906, **74**, [2], 226.

Naunton, *J. Soc. Chem. Ind.*, 1926, **45**, 377T.

***N*-Phenyl-*N'*-*m*-tolylthiourea (m-Methylthiocarbanilide).**

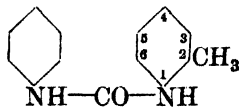
Prisms from EtOH. M.p. 91–2°. Very sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. AgNO<sub>3</sub> in EtOH → phenyl-*m*-tolylurea.

Dixon, *J. Chem. Soc.*, 1895, **67**, 557.

***N*-Phenyl-*N'*-*p*-tolylthiourea (p-Methylthiocarbanilide).**

M.p. 142°. Very sol. EtOH. Sol. Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Hydrazine → 4-*p*-tolylthiosemicarbazide. Phenylhydrazine → 1-phenyl-4-*p*-tolylthiosemicarbazide.

Gebhardt, *Ber.*, 1884, **17**, 3035.

***N*-Phenyl-*N'*-*o*-tolylurea (o-Methylcarbanilide)** $C_{14}H_{14}ON_2$ 

MW, 226

Needles from EtOH. M.p. 212° (196°).

Sonn, *Ber.*, 1914, **47**, 2442.

***N*-Phenyl-*N'*-*m*-tolylurea (m-Methylcarbanilide).**

Needles from EtOH. M.p. 173–4°.

Buchka, Schachtebeck, *Ber.*, 1889, **22**, 840.

***N*-Phenyl-*N'*-*p*-tolylurea (p-Methylcarbanilide).**

Needles from EtOH. M.p. 226°. Spar. sol. cold EtOH.

Mayer, *Bull. soc. chim.*, 1916, **19**, 430.

**1-Phenyl-1 : 2 : 3-triazole** $C_8H_7N_3$ 

MW, 145

Plates from warm H<sub>2</sub>O. M.p. 56°. B.p. 172–4°/18.5 mm.

Bertho, *Ber.*, 1925, **58**, 862.

Dimroth, Fester, *Ber.*, 1910, **43**, 2222.

**1-Phenyl-1 : 2 : 4-triazole** $C_8H_7N_3$ 

MW, 145

Needles. M.p. 47°. B.p. 266°. Sol. EtOH. *B, HNO*<sub>3</sub>: needles. M.p. 141°. Spar. sol. cold H<sub>2</sub>O.

*B*<sub>2</sub>*H*<sub>3</sub>*PtCl*<sub>6</sub>: yellow needles + 3H<sub>2</sub>O from HCl. Also orange-red prisms + 2H<sub>2</sub>O. Boiling H<sub>2</sub>O → *B*<sub>2</sub>*PtCl*<sub>4</sub>, yellow powder. Insol. H<sub>2</sub>O, HCl.

*Picrate*: cryst. from H<sub>2</sub>O. M.p. 159°.

Pellizzari, *Gazz. chim. ital.*, 1911, **41**, ii, 20.

Widman, *Ber.*, 1893, **26**, 2615.

**3-Phenyl-1 : 2 : 4-triazole.**

Needles. M.p. 119.5–120°.

*B, HCl*: needles. M.p. 195°.

*Acetyl*: m.p. 90°.

*B*<sub>2</sub>*H*<sub>3</sub>*PtCl*<sub>6</sub>: yellow plates + 2H<sub>2</sub>O from dil. HCl. Decomp. at 255°.

See first reference above and also

Young, Oates, *J. Chem. Soc.*, 1901, **79**, 665.

**1-Phenyl-1 : 2 : 5-triazole** $C_8H_7N_3$ 

MW, 145

B.p. 223–4°/716 mm. Spar. sol. H<sub>2</sub>O. Misc. with EtOH.

Jonas, Pechmann, *Ann.*, 1891, **262**, 290.

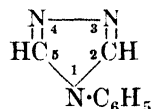
**3-Phenyl-1 : 2 : 5-triazole.**

Scales from EtOH. Aq. or C<sub>6</sub>H<sub>6</sub>. M.p. 143–5°. Exhibits acidic and basic properties.

*B, HCl*: cryst. powder. M.p. 140°. Insol. cold  $H_2O$ .

Oliveri-Mandalà, Coppola, *Chem. Zentr.*, 1910, II, 225.

## 1-Phenyl-1 : 3 : 4-triazole



$C_8H_7N_3$

MW, 145

Prisms from  $H_2O$ , needles from  $C_6H_6$ . M.p. 122°. Sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ . Decomp. on dist.  $KMnO_4 \rightarrow$  1 : 3 : 4-triazole.  $CuSO_4 \rightarrow$  light blue ppt.  $AgNO_3$  and  $HgCl_2 \rightarrow$  white ppts.

$B_2H_2PtCl_6$ : needles from HCl.

*Picrate*: needles from EtOH. M.p. 172°.

Heller, Köhler, Gottfried, Arnold, Herrmann, *J. prakt. Chem.*, 1928, 120, 62.

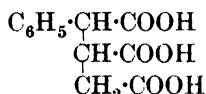
Pellizzari, Massa, *Atti accad. Lincei*, 1901, 10, I, 366.

## 5-Phenyl-1 : 3 : 4-triazole.

Pale yellow plates from EtOH.Aq. M.p. 177°.

De, Roy-Choudhury, *J. Indian Chem. Soc.*, 1928, 5, 275.

## 1-Phenyltricarballic Acid



$C_{12}H_{12}O_6$

MW, 252

Two forms.

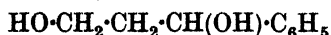
(i) Needles from  $Me_2CO$  or  $C_6H_6$ , scales from  $H_2O$ . M.p. 199° decomp. Very sol. hot  $H_2O$ ,  $Me_2CO$ . Spar. sol.  $CHCl_3$ , boiling  $C_6H_6$ ,  $CS_2$ .

(ii) Prisms +  $1H_2O$  from warm  $H_2O$ . M.p. 110° (in closed capillary, m.p. 110–15°, then solidifies and remelts at 196–201°).

Wegscheider, *Ber.*, 1911, 44, 908.

Stobbe, Fischer, *Ann.*, 1901, 315, 231.

Hecht, *Monatsh.*, 1903, 24, 370.

1-Phenyltrimethylene Glycol (1-Phenylpropandiol-1 : 3,  $\alpha\gamma$ -dihydroxypropylbenzene)

$C_9H_{12}O_2$

MW, 152

Colourless odourless oil. B.p. 175°/11 mm. (165°/10 mm.).

*Dibenzoyl*: needles from ligroin. M.p. 51°.

*Di-p-nitrobenzoyl*: m.p. 110–110.5°.

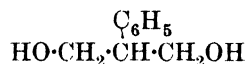
Dict. of Org. Comp.—III.

*Di-Me ether*:  $C_{11}H_{16}O_2$ . MW, 180. B.p. 215–17° part. decomp., 94–5°/15 mm., 89–91°/12 mm.  $D_4^{20}$  0.983.

Rupe, Müller, *Helv. Chim. Acta*, 1921, 4, 844.

St. Pfau, Plattner, *Helv. Chim. Acta*, 1932, 15, 1265.

Straus, Berkow, *Ann.*, 1913, 401, 155.

2-Phenyltrimethylene Glycol (2-Phenylpropandiol-1 : 3,  $\beta\beta'$ -dihydroxyisopropylbenzene)

$C_9H_{12}O_2$

MW, 152

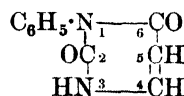
Viscous oil. B.p. 176°/13 mm.  $D_4^{19}$  1.1161.  $n_D^{19}$  1.54267.

*Diacetyl*: b.p. 162–4°/13 mm.

*Methylene ether*: b.p. 128–30°/13 mm.  $D_4^{18}$  1.1111.  $n_D^{18}$  1.53063.

Prins, *Chem. Abstracts*, 1920, 14, 1119, 1662.

## 1-Phenyluracil



$C_{10}H_8O_2N_2$

MW, 188

Cryst. from  $H_2O$ . M.p. 247°. Spar. sol.  $H_2O$ , MeOH, EtOH, Py. Insol.  $Et_2O$ , AcOEt,  $Me_2CO$ ,  $CHCl_3$ , ligroin,  $C_6H_6$ .

Buerger, Johnson, *J. Am. Chem. Soc.*, 1934, 56, 2755.

## 4-Phenyluracil.

Microscopic needles and prisms from EtOH or AcOH.Aq. M.p. 269–70° decomp. Sol. 100 parts  $H_2O$  at 100°, 35 parts EtOH at 78°. Sol. AcOH, KOH. Spar. sol.  $Et_2O$ . Stable to HCl,  $NH_2 \cdot NH_2$ ,  $CH_3I$ .

*K salt*: cryst. from  $H_2O$ . Decomp. above 300°.

*1-Me*: plates from EtOH. M.p. 228–30°. Sol. boiling EtOH, boiling  $CHCl_3$ .

*1 : 3-Di-Me*: plates from EtOH. M.p. 122–122.5°. Sol. boiling EtOH,  $CHCl_3$ .

Evans, Johnson, *J. Am. Chem. Soc.*, 1930, 52, 4999.

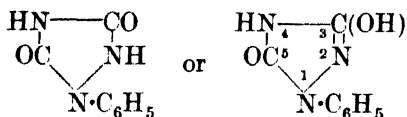
Johnson, Hemingway, *J. Am. Chem. Soc.*, 1915, 37, 379.

Warmington, *J. prakt. Chem.*, 1893, 47, 203.

**5-Phenyluracil.**

Microscopic plates from  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . Does not melt below  $350^\circ$ . Sol. alkalis. Spar. sol. hot  $\text{H}_2\text{O}$ , hot  $\text{EtOH}$ .

Wheeler, Bristol, *Am. Chem. J.*, 1904, **33**, 448.

**1-Phenylurazole**

$\text{C}_8\text{H}_7\text{O}_2\text{N}_3$  MW, 177

Plates from  $\text{H}_2\text{O}$ . M.p.  $263-4^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , alkalis. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Behaves as monobasic acid. Reddens litmus.  $\text{CO}_2$  liberated from carbonates. Stable to boiling alk. sols.  $\text{FeCl}_3 \rightarrow$  violet col. *O*-acyl and *O*-alkyl comps. with  $\text{HCl} \rightarrow$  *N*-comps.  $\text{PCl}_5 \rightarrow$  1-phenyl-3:5-dichlorotriazole.

*Ag salt*: m.p.  $252^\circ$  decomp. Very sol.  $\text{NH}_4\text{OH}$ . Insol. dil.  $\text{HNO}_3$ .

*Ba salt*: plates +  $2\text{H}_2\text{O}$ . Decomp. at  $280-300^\circ$ .

2-*Me*:  $\text{C}_9\text{H}_9\text{O}_2\text{N}_3$ . MW, 191. Needles. M.p.  $183^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , alkalis. Spar. sol.  $\text{H}_2\text{O}$ . Stable to  $\text{H}_2\text{SO}_4$  at  $100^\circ$ . *Ag salt*: m.p.  $250^\circ$  decomp. Insol. dil.  $\text{HNO}_3$ . Very sol.  $\text{NH}_4\text{OH}$ .

4-*Me*: m.p.  $225^\circ$ . Sol.  $\text{CHCl}_3$ . 2-*Acetyl*: needles from  $\text{EtOH}$ . M.p.  $94-5^\circ$ . 2-*Benzoyl*: cryst. M.p.  $185^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ . 3-*Me ether*: needles from alk.  $\text{EtOH.Aq.}$  M.p.  $95^\circ$ . 3-*Et ether*: cryst. from alk.  $\text{EtOH.Aq.}$  M.p.  $95^\circ$ .

2:4-*Di-Me*:  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{N}_3$ . MW, 205. Prisms or plates from  $\text{EtOH.Aq.}$ , or ligroin. M.p.  $95^\circ$ . Very sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{Et}_2\text{O}$ .  $\text{NaOH} \rightarrow$  1-phenyl-2:4-dimethylsemicarbazide and 1-phenyl-2-methylurazole.

2-*Et*:  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{N}_3$ . MW, 205. M.p.  $119^\circ$ . Very sol. hot  $\text{H}_2\text{O}$ , hot  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. sol. cold  $\text{H}_2\text{O}$ , cold  $\text{EtOH}$ . *Ag salt*: m.p.  $239^\circ$  decomp.

2-*Me*-4-*Et*:  $\text{C}_{11}\text{H}_{13}\text{O}_2\text{N}_3$ . MW, 219. Cryst. from alk.  $\text{EtOH.Aq.}$  M.p.  $113^\circ$ .

3-*Me ether*:  $\text{C}_9\text{H}_9\text{O}_2\text{N}_3$ . MW, 191. Plates. M.p.  $197^\circ$ . Gives mono K salt.  $\text{HCl} \rightarrow$  phenylurazole.

3(5)-*Et ether*:  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{N}_3$ . MW, 205. Two forms. May be 3- and 5-*Et ethers*. (i) M.p.  $141^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ , alkalis. Spar. sol. ligroin.  $\text{EtOH-HCl} \rightarrow$  phenylurazole. (ii) Needles from  $\text{EtOH}$ .

M.p.  $152^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , alkalis. Behaves as monobasic acid.

3:5-*Di-Et ether*:  $\text{C}_{12}\text{H}_{15}\text{O}_2\text{N}_3$ . MW, 233. Needles from  $\text{EtOH.Aq.}$  M.p.  $53^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ .  $\text{HCl} \rightarrow$  phenylurazole.

2-*Acetyl*: needles from  $\text{H}_2\text{O}$ , plates from  $\text{C}_6\text{H}_6$ . M.p.  $175^\circ$ .

2:4-*Diacetyl*: needles from  $\text{C}_6\text{H}_6$ . M.p.  $169^\circ$ . Insol.  $\text{H}_2\text{O}$ .  $\text{EtOH} \rightarrow$  2-acetyl.

2:4-*Dibenzoyl*: m.p.  $178-80^\circ$ .

Murray, Dains, *J. Am. Chem. Soc.*, 1934, **56**, 144.

Dains, Wertheim, *J. Am. Chem. Soc.*, 1920, **42**, 2308.

Brunel, Acree, *Am. Chem. J.*, 1912, **43**, 505.

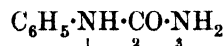
Acree, *Am. Chem. J.*, 1907, **38**, 1; *Ber.*, 1903, **36**, 3139; *Ber.*, 1902, **35**, 557.

**4-Phenylurazole.**

Prisms from  $\text{H}_2\text{O}$ . M.p.  $203^\circ$ . Sol.  $\text{EtOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Oxidising agents  $\rightarrow$  deep red col.

*N-Me*: cryst. M.p.  $188^\circ$ . Very sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Strongly acidic.

Thiele, Stange, *Ann.*, 1894, **283**, 45.

**Phenylurea**

$\text{C}_7\text{H}_8\text{ON}_2$  MW, 136

Needles or plates from  $\text{H}_2\text{O}$ , tablets from  $\text{EtOH}$ . M.p.  $147^\circ$ . Sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Heat of comb.  $\text{C}_7$  880 Cal. Heat  $\rightarrow$  *N*:*N'*-diphenylurea.  $\text{H}_2\text{SO}_4 \rightarrow$  sulphanilic and sulphocarbanilic acids.  $\text{NaNO}_2 \rightarrow$  *N*-nitroso-*N*-phenylurea. Hydrazine  $\rightarrow$  4-phenylsemicarbazide.  $\text{NaOEt} \rightarrow$  mono-Na deriv.

*B,HCl*: plates. M.p.  $114-16^\circ$  decomp. Decomp. by  $\text{H}_2\text{O}$ .

*B,HNO}\_3*: plates. M.p.  $134-5^\circ$  decomp. Decomp. by  $\text{H}_2\text{O}$ .

*B}\_2\text{,H}\_2\text{AuCl}\_4*: red needles. M.p.  $147^\circ$ . Decomp. by  $\text{H}_2\text{O}$ .

*B}\_3\text{,H}\_2\text{PtCl}\_6*: orange cryst. M.p.  $173-5^\circ$  decomp.

1-*N-Acetyl*: needles from hot  $\text{H}_2\text{O}$ . M.p.  $167^\circ$ .

2-*N-Acetyl*: see sym.-Acetylphenylurea.

2-*N-Benzoyl*: needles from  $\text{EtOH}$ . M.p.  $210^\circ$  ( $202^\circ$ ). Spar. sol. cold  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

1-*N-Nitroso*:  $\text{C}_6\text{H}_5\text{N}(\text{NO})\cdot\text{CO}\cdot\text{NH}_2$ .  $\text{C}_7\text{H}_7\text{O}_2\text{N}_3$ . MW, 165. Yellow needles from  $\text{Et}_2\text{O-pet.}$

ether. M.p. 95° decomp. Sol. EtOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Palit, *J. Indian Chem. Soc.*, 1934, **11**, 479.

Mauguin, *Ann. chim. phys.*, 1911, **22**, 346.

Pickard, Kenyon, *J. Chem. Soc.*, 1907, **91**, 902.

Schiff, *Ann.*, 1907, **352**, 83.

Stieglitz, Earle, *Am. Chem. J.*, 1903, **30**, 418.

Hoffmann, *Ann.*, 1849, **70**, 130.

Doht, Haager, *Monatsh.*, 1903, **24**, 853.

**Phenylurethane** (*Phenylcarbamic ethyl ester, carbanilic acid ethyl ester*)

C<sub>9</sub>H<sub>11</sub>O<sub>2</sub>N      C<sub>6</sub>H<sub>5</sub>·NH·CO·OC<sub>2</sub>H<sub>5</sub>      MW, 165

Needles from H<sub>2</sub>O, plates from EtOH.Aq. M.p. 53°. B.p. 237° (slight decomp.), 152°/14 mm. Very sol. EtOH, Et<sub>2</sub>O. Insol. cold H<sub>2</sub>O. Heat of comb. C<sub>p</sub> 1128.3 Cal. EtOH sol. with AgNO<sub>3</sub> + drop of NaOH → deep red to black col. NH<sub>3</sub> → aniline + urea. P<sub>2</sub>O<sub>5</sub> → phenyl isocyanate. R·NH<sub>2</sub> → C<sub>6</sub>H<sub>5</sub>·NH·CO·NH·R.

*K deriv.*: needles. Decomp. by H<sub>2</sub>O.

*N-Acetyl*: cryst. from ligroin. M.p. 59°. B.p. 142°/10 mm.

*N-Benzoyl*: m.p. 160–1°.

*N-Nitroso*: C<sub>9</sub>H<sub>10</sub>O<sub>3</sub>N<sub>2</sub>. MW, 194. Pale yellow needles from pet. ether. M.p. 61–2°. Sol. EtOH, Et<sub>2</sub>O, AcOH.

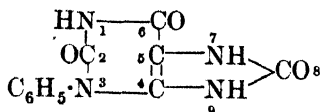
Binaghi, *Gazz. chim. ital.*, 1932, **62**, 469.

Nekrassow, Melnikow, *J. prakt. Chem.*, 1930, **126**, 92.

Weizmann, Garrard, *J. Chem. Soc.*, 1920, **117**, 328.

Nijk, *Rec. trav. chim.*, 1920, **39**, 700.

### 3-Phenyluric Acid



C<sub>11</sub>H<sub>8</sub>O<sub>3</sub>N<sub>4</sub>      MW, 244

Cryst. + 1H<sub>2</sub>O from EtOH.Aq. or AcOH.Aq. Decomp. at 270–300°. Sol. hot EtOH, AcOH. Spar. sol. cold H<sub>2</sub>O.

*1-Me*: C<sub>12</sub>H<sub>10</sub>O<sub>3</sub>N<sub>4</sub>. MW, 258. Yellowish needles from AcOH. Does not melt below 340°.

*1:7:9-Tri-Me*: C<sub>14</sub>H<sub>14</sub>O<sub>3</sub>N<sub>4</sub>. MW, 286. Cryst. from H<sub>2</sub>O. M.p. 229°.

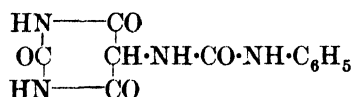
Hepner, Frenkenberg, *Helv. Chim. Acta*, 1932, **15**, 536.

### 9-Phenyluric Acid.

Plates + 2H<sub>2</sub>O from H<sub>2</sub>O. Sol. 120 parts H<sub>2</sub>O at 100°. Anhyd. at 130°. Decomp. at 320°. Spar. sol. EtOH. Sol. warm H<sub>2</sub>SO<sub>4</sub>. Reduces NH<sub>3</sub>·AgNO<sub>3</sub>. Gives murexide reaction.

Fischer, *Ber.*, 1900, **33**, 1704.

### Phenyl-ψ-uric Acid



C<sub>11</sub>H<sub>10</sub>O<sub>4</sub>N<sub>4</sub>      MW, 262

Aggregates of microscopic needles from H<sub>2</sub>O. Contains H<sub>2</sub>O of cryst. Contains ½H<sub>2</sub>O at 120°, chars above this temp. Sol. 350 parts H<sub>2</sub>O. Reduces NH<sub>3</sub>·AgNO<sub>3</sub>. Gives murexide reaction. HCl → 9-phenylurea. Na, K and NH<sub>4</sub> salts spar. sol. cold H<sub>2</sub>O.

Fischer, *Ber.*, 1900, **33**, 1703.

### 1-Phenylvaleraldehyde (α-Phenyl-n-valeraldehyde)

C<sub>11</sub>H<sub>14</sub>O      C<sub>6</sub>H<sub>5</sub>  
CH<sub>3</sub>·CH<sub>2</sub>·CH<sub>2</sub>·CH·CHO      MW, 162

Oil. B.p. 122–3°/28 mm.

*Semicarbazone*: m.p. 115–16°.

Darzens, *Compt. rend.*, 1904, **139**, 1216.

### 4-Phenylvaleraldehyde

C<sub>11</sub>H<sub>14</sub>O      C<sub>6</sub>H<sub>5</sub>·CH<sub>2</sub>·CH<sub>2</sub>·CH<sub>2</sub>·CH<sub>2</sub>·CHO      MW, 162

Oil with lemon odour. B.p. 129–31°/10 mm.

*p-Nitrophenylhydrazone*: pale yellow cryst. powder from EtOH.Aq. M.p. 82–4°.

*Di-Me acetal*: C<sub>13</sub>H<sub>20</sub>O<sub>2</sub>. MW, 208. B.p. 136–9°/11 mm.

v. Braun, Kruber, *Ber.*, 1912, **45**, 399.

### 1-Phenylvaleric Acid (α-Phenyl-n-valeric acid, 1-phenylbutane-1-carboxylic acid, propyl-phenylacetic acid)

C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>      C<sub>6</sub>H<sub>5</sub>  
CH<sub>3</sub>·CH<sub>2</sub>·CH<sub>2</sub>·CH·COOH      MW, 178

*d.*

Viscous oil. B.p. 165°/14 mm. D<sub>4</sub><sup>20</sup> 1.047. [α]<sub>D</sub><sup>20</sup> + 72.10°, [α]<sub>D</sub><sup>20</sup> + 58.81° in CHCl<sub>3</sub>, [α]<sub>D</sub><sup>20</sup> + 33.1° in Et<sub>2</sub>O.

*l.*

[α]<sub>D</sub><sup>20</sup> – 14.1° in Et<sub>2</sub>O.

*dl.*

Needles from ligroin. M.p. 52°. B.p. 280°. Resolved into active components with *l*-menthylamine.

*Amide*:  $C_{11}H_{15}ON$ . MW, 177. Cryst. from EtOH.Aq. M.p. 83–5°. Sol. org. solvents. Spar. sol.  $H_2O$ .

*Nitrile*:  $C_{11}H_{13}N$ . MW, 159. B.p. 254–5°/750 mm., 125–8°/13 mm.  $D_{15}^{20}$  0.960. Immiscible with  $H_2O$ . Misc. with EtOH,  $C_6H_6$ . Volatile in steam.

Levene, Marker, *J. Biol. Chem.*, 1930, **88**, 53.

Bayer, D.R.P., 249,241, (*Chem. Zentr.*, 1912, II, 396).

Pickard, Yates, *J. Chem. Soc.*, 1909, **95**, 1017.

**2-Phenylvaleric Acid** ( $\beta$ -Phenyl-*n*-valeric acid,  $\beta$ -ethylhydrocinnamic acid, 2-phenylbutane-1-carboxylic acid)

$$C_{11}H_{14}O_2 \quad \begin{array}{c} C_6H_5 \\ | \\ CH_3 \cdot CH_2 \cdot CH \cdot CH_2 \cdot COOH \end{array} \quad MW, 178$$

*d.*

B.p. 142°/5 mm.  $[\alpha]_D^{25} + 2.86^\circ$  in  $C_6H_6$ .

*l.*

B.p. 140°/4 mm.  $[\alpha]_D^{25} - 16.5^\circ$  in  $C_6H_6$ .

*Et ester*:  $C_{13}H_{18}O_2$ . MW, 206. B.p. 110°/2 mm.  $[\alpha]_D^{25} - 7.41^\circ$ .

*dl.*

Cryst. from Et<sub>2</sub>O. M.p. 66°. Very sol. EtOH, Et<sub>2</sub>O.

*Me-anilide*: b.p. 206°/12 mm.

*Et-anilide*: b.p. 214°/15 mm.

Maxim, Ioanid, *Chem. Zentr.*, 1928, II, 755.

Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 333; 1932, **97**, 388; 1931, **93**, 763.

**3-Phenylvaleric Acid** ( $\gamma$ -Phenyl-*n*-valeric acid, 3-phenylbutane-1-carboxylic acid)

$$C_{11}H_{14}O_2 \quad \begin{array}{c} C_6H_5 \\ | \\ CH_3 \cdot CH \cdot CH_2 \cdot CH_2 \cdot COOH \end{array} \quad MW, 178$$

*l.*

B.p. 137°/1 mm.  $[\alpha]_D^{20} - 1.06^\circ$ .

*Et ester*:  $C_{13}H_{18}O_2$ . MW, 206. B.p. 112°/1 mm.  $[\alpha]_D^{25} - 1.23^\circ$ .

*dl.*

Cryst. M.p. 13°. B.p. 210°/85 mm., 170°/10 mm., 147°/1 mm.  $D_{15}^{20}$  1.0554.

*Chloride*:  $C_{11}H_{13}OCl$ . MW, 196.5. B.p. 811–19°/13 mm.

*Nitrile*:  $C_{11}H_{13}N$ . MW, 159. B.p. 125–6°/13 mm.

Levene, Marker, *J. Biol. Chem.*, 1935, **110**, 338.

Braun, Stuckenschmidt, *Ber.*, 1923, **56**, 1727.

Mayer, Stamm, *ibid.*, 1431.

Eijkmann, *Chem. Zentr.*, 1904, I, 1416.

**4-Phenylvaleric Acid** ( $\delta$ -Phenyl-*n*-valeric acid, 4-phenylbutane-1-carboxylic acid)

$$C_{11}H_{14}O_2 \quad C_6H_5 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot COOH \quad MW, 178$$

Plates from hot  $H_2O$ , prisms from pet. ether. M.p. 57° (61°). Sol. ord. org. solvents. Spar. sol. hot  $H_2O$ .

*Me ester*:  $C_{12}H_{16}O_2$ . MW, 192. B.p. 173°/35 mm.

*Et ester*:  $C_{13}H_{18}O_2$ . MW, 206. B.p. 150°/11 mm.

*Amide*:  $C_{11}H_{15}ON$ . MW, 177. M.p. 109°.

*Nitrile*:  $C_{11}H_{13}N$ . MW, 159. B.p. 157–61°/17 mm.

*Anilide*: plates from MeOH. M.p. 89–90°.

Staudinger, Müller, *Ber.*, 1923, **56**, 713.

Rupe, *Ann.*, 1909, **369**, 343.

v. Braun, Deutsch, *Ber.*, 1912, **45**, 2178.

**4-Phenyl- $\gamma$ -valerolactone.**

See under 3-Hydroxy-4-phenyl-*n*-valeric Acid.

**N-Phenylvaline.**

See 1-Anilinoisovaleric Acid.

**3-Phenylvinylacetic Acid.**

See Styrylacetic Acid.

**Phenylvinylacrylic Acid.**

See Styrylacrylic Acid.

**1-Phenylvinyl Alcohol** ( $\alpha$ -Hydroxystyrene)

$$C_8H_8O \quad CH_2 \cdot C(OH) \cdot C_6H_5 \quad MW, 120$$

*Me ether*:  $C_9H_{10}O$ . MW, 134. Oil with aromatic odour. B.p. 197°.  $D_4^{21}$  1.003.  $n_D^{25}$  1.5400. Dil.  $H_2SO_4 \rightarrow$  acetophenone. Semicarbazide acetate  $\rightarrow$  semicarbazone of acetophenone.

*Et ether*:  $C_{10}H_{12}O$ . MW, 148. Oil. B.p. 209–10°, 88–9°/11 mm.  $D_4^{20}$  0.9709.  $n_D^{17.5}$  1.5304. Alc. HCl  $\rightarrow$  acetophenone.

*Isoamyl ether*:  $C_{13}H_{18}O$ . MW, 190. B.p. 255–9°.  $D_4^{20}$  0.943.

*Phenyl ether*:  $C_{14}H_{14}O$ . MW, 196. B.p. 151°/14 mm.  $D_{17}^{20}$  1.10729.

Auwers, *Ber.*, 1911, **44**, 3520.

Moureu, *Compt. rend.*, 1903, **137**, 261.

Tiffeneau, *Compt. rend.*, 1907, **145**, 813.

**2-Phenylvinyl Alcohol** ( $\beta$ -Hydroxystyrene) $\text{C}_8\text{H}_8\text{O}$ 

MW, 120

*Me ether*: oil. B.p. 210–13°, 99°/13 mm.  $D_4^{23.3}$  0.9894.  $n_D^{24.3}$  1.5620. Dil.  $\text{H}_2\text{SO}_4 \rightarrow$  phenylacetaldehyde.

*Et ether*: oil with aromatic odour. B.p. 223–6°, 105°/14 mm.  $D_4^{21.4}$  0.9714.  $n_D^{21.2}$  1.5502. Forms unstable add. comp. with Br. Boiling  $\text{H}_2\text{O} \rightarrow$  phenylacetaldehyde.

*Propyl ether*:  $\text{C}_{11}\text{H}_{14}\text{O}$ . MW, 162. B.p. 238–41°.  $D_0^{15.5}$  0.966.  $n_D^{15.5}$  1.542.

*Isobutyl ether*:  $\text{C}_{12}\text{H}_{16}\text{O}$ . MW, 176. B.p. 248–51°.  $D_0^{16}$  0.946.  $n_D^{16}$  1.5342.

*Phenyl ether*: b.p. 180°/16 mm., 157–8°/7 mm.  $\text{Na} + \text{EtOH} \rightarrow$  ethylbenzene.  $\text{HI} \rightarrow$  2-phenylnaphthalene. Forms unstable bromine add. comp. Acid hyd.  $\rightarrow$  phenylacetaldehyde.

*Acetyl*: two stereoisomeric forms. *Cis*: b.p. 68°/0 mm.  $D_0^{20}$  1.073.  $n_D^{20}$  1.5553. *Trans*: b.p. 68°/0 mm.  $D_0^{20}$  1.065.  $n_D^{20}$  1.5518. Red.  $\rightarrow$  2-phenylethyl alcohol. Catalytic red.  $\rightarrow$  2-phenylethyl acetate. Hyd.  $\rightarrow$  phenylacetaldehyde.

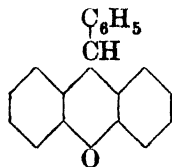
Böeseken, Kremer, *Rec. trav. chim.*, 1931, 50, 830.

Späth, *Monatsh.*, 1915, 36, 6.

Auwers, Eisenlohr, *J. prakt. Chem.*, 1910, 82, 100.

Stoermer, Biesenbach, *Ber.*, 1905, 38, 1961.

Moureu, *Compt. rend.*, 1903, 137, 288.

**5-Phenylxanthene** $\text{C}_{19}\text{H}_{14}\text{O}$ 

MW, 258

Cryst. from boiling EtOH. M.p. 145°. Sol.  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, AcOH, ligroin. Insol. cold  $\text{H}_2\text{SO}_4$ .

Ruszig, *Z. angew. Chem.*, 1919, 32, 39.

Baeyer, *Ann.*, 1907, 354, 170.

**Phenyl xenyl Ketone.**

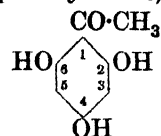
See Phenylbenzophenone.

**Phenyl-xylene.**

See Dimethyldiphenyl.

**Phenyl p-xylyl Ketone.**

See 4-Methyldeoxybenzoin.

**Phloracetophenone** (2 : 4 : 6-Trihydroxyacetophenone, acetophloroglucinol) $\text{C}_8\text{H}_8\text{O}_4$ 

MW, 168

Needles from  $\text{H}_2\text{O}$ . M.p. anhyd. 219°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ , AcOH. Spar. sol.  $\text{CHCl}_3$ , hot  $\text{C}_6\text{H}_6$ .

*2-Me ether*:  $\text{C}_9\text{H}_{10}\text{O}_4$ . MW, 182. M.p. 205–7°.

*4-Me ether*: m.p. 136–7° (139–40°).

*2 : 4-Di-Me ether*:  $\text{C}_{10}\text{H}_{12}\text{O}_4$ . MW, 196. Constituent of fruit of *Xanthoxylum alatum*, Roxb., and volatile oil of *X. aubertia*, DC. Cryst. from EtOH. M.p. 85–8°. Sol. EtOH,  $\text{Et}_2\text{O}$ , alkalis. Insol.  $\text{H}_2\text{O}$ . *Oxime*: m.p. 108–10°. *Acetyl*: prisms from EtOH. M.p. 107°.

*2 : 6-Di-Me ether*:  $\text{C}_{10}\text{H}_{12}\text{O}_4$ . MW, 196. Prisms from AcOEt. M.p. 185.5°. *4-Benzoyl*: m.p. 119°.

*Tri-Me ether*:  $\text{C}_{11}\text{H}_{14}\text{O}_4$ . MW, 210. Prisms. M.p. 103° (100°).

*2-Me-4-Et ether*:  $\text{C}_{11}\text{H}_{14}\text{O}_4$ . MW, 210. M.p. 56–7°.

*4-Me-2-Et ether*: m.p. 133–4°.

*2 : 4-Di-Me-6-Et ether*:  $\text{C}_{12}\text{H}_{16}\text{O}_4$ . MW, 224. M.p. 69–70°.

*2 : 6-Di-Me-4-Et ether*: m.p. 81–2°.

*2 : 4-Di-Et ether*:  $\text{C}_{12}\text{H}_{16}\text{O}_4$ . MW, 224. Needles from EtOH.Aq. M.p. 85°.

*Tri-Et ether*:  $\text{C}_{14}\text{H}_{20}\text{O}_4$ . MW, 252. Plates from EtOH.Aq. M.p. 75°.

*Triacetyl*: m.p. 103°.

*2-Benzoyl*: m.p. 168°.

*4-Benzoyl*: m.p. 210–11°.

*Tribenzoyl*: m.p. 117–18°.

Gulati, Seth, Venkataraman, *J. Chem. Soc.*, 1934, 1765; *Organic Syntheses*, 1935, XV, 70.

Tseng, *Chem. Zentr.*, 1935, II, 2826.

Sonn, *Ber.*, 1928, 61, 2300.

Shinoda, Sato, *Chem. Abstracts*, 1928, 22, 2947.

Howells, Little, *J. Am. Chem. Soc.*, 1932, 54, 2452.

Kostanecki, Tambor, *Ber.*, 1899, 32, 2262.

**Phloramine.**

See 5-Aminoresorcinol.

**Phloraspin** $\text{C}_{23}\text{H}_{28}\text{O}_8$ 

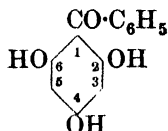
MW, 432

Constituent of *Filix mas* extract. Needles from  $\text{Me}_2\text{CO}$ .Aq. M.p. 211°. Mod. sol.  $\text{CHCl}_3$ .

$\text{Me}_2\text{CO}$ ,  $\text{AcOEt}$ ,  $\text{AcOH}$ , hot  $\text{EtOH}$ , hot xylene. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether, ligroin.  $\text{FeCl}_3 \rightarrow$  reddish-brown col. in  $\text{EtOH}$ . Sol. alkalis or conc.  $\text{H}_2\text{SO}_4 \rightarrow$  butyric acid. Possibly identical with flavaspidin, *q.v.*

Boehm, *Ann.*, 1903, 329, 338.

**Phlorbenzophenone** (2 : 4 : 6-Trihydroxybenzophenone)



$\text{C}_{13}\text{H}_{10}\text{O}_4$

MW, 230

Yellow needles +  $1\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $165^\circ$ .  $\text{NaOH} \rightarrow$  red col.  $\text{FeCl}_3 \rightarrow$  brownish-red col. Reduces  $\text{NH}_3$ ,  $\text{AgNO}_3$  and Fehling's. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{AcOEt}$ . Spar. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin.

2-Me ether: see Isocotoin.

4-Me ether: see Cotoin.

2 : 4-Di-Me ether:  $\text{C}_{15}\text{H}_{14}\text{O}_4$ . MW, 258.

6-Acetyl: prisms from  $\text{EtOH}$ . M.p.  $83^\circ$ .

2 : 6-Di-Me ether: plates from  $\text{EtOH.Aq.}$  M.p.  $178-9^\circ$ . 4-Benzoyl: prisms from  $\text{EtOH}$ . M.p.  $172^\circ$ .

4-Me-2 : 6-Di-Et ether:  $\text{C}_{18}\text{H}_{20}\text{O}_4$ . MW, 300. M.p.  $82-3^\circ$ .

2 : 4-Di-Me-6-Et ether:  $\text{C}_{17}\text{H}_{18}\text{O}_4$ . MW, 286. M.p.  $103-4^\circ$ .

4-Benzoyl: needles from  $\text{C}_6\text{H}_6$ . M.p.  $186^\circ$ .

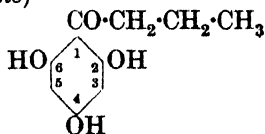
2 : 4 : 6-Tribenzoyl: prisms from  $\text{MeOH}$ . M.p.  $125-6^\circ$  ( $172^\circ$ ).

Canter, Curd, Robertson, *J. Chem. Soc.*, 1931, 1254.

Hoesch, *Ber.*, 1915, 48, 1131.

Klarmann, Figdor, *J. Am. Chem. Soc.*, 1926, 48, 804.

**Phlorbutyrophenone** (2 : 4 : 6-Trihydroxybutyrophenone)



$\text{C}_{10}\text{H}_{12}\text{O}_4$

MW, 196

Cryst. +  $1\text{H}_2\text{O}$ . M.p.  $183^\circ$  anhyd. ( $181^\circ$ ).

(?) 2-Me ether:  $\text{C}_{11}\text{H}_{14}\text{O}_4$ . MW, 210. Needles. M.p.  $130^\circ$ .

(?) 4-Me ether: leaflets. M.p.  $113^\circ$ .

2 : 4-Di-Me ether:  $\text{C}_{12}\text{H}_{16}\text{O}_4$ . MW, 224. Prisms from  $\text{EtOH.Aq.}$  M.p.  $70^\circ$ .

2 : 6-Di-Me ether: prisms from  $\text{AcOEt}$ . M.p.  $107^\circ$ . 4-Benzoyl: m.p.  $86^\circ$ .

4-Benzoyl: m.p.  $164^\circ$ .

Canter, Curd, Robertson, *J. Chem. Soc.*, 1931, 1252.

**Phloretic Acid** (Phloretinic acid, p-hydrocoumaric acid, 4-hydroxyhydrocinnamic acid)



$\text{C}_9\text{H}_{10}\text{O}_3$

MW, 166

Occurs in human urine. Prisms from  $\text{Et}_2\text{O}$ . M.p.  $129-30^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Insol.  $\text{CS}_2$ .  $k = 1.73 \times 10^{-5}$  at  $25^\circ$  ( $2.03 \times 10^{-5}$ ). No col. with  $\text{FeCl}_3$ .

Me ester:  $\text{C}_{10}\text{H}_{12}\text{O}_3$ . MW, 180. Cryst. from  $\text{Et}_2\text{O}$ -pet. ether. M.p.  $40-1^\circ$ . B.p.  $186-7^\circ/17$  mm.

Et ester:  $\text{C}_{11}\text{H}_{14}\text{O}_3$ . MW, 194. M.p.  $45^\circ$ . B.p.  $193^\circ/18$  mm.

Amide:  $\text{C}_9\text{H}_{11}\text{O}_2\text{N}$ . MW, 165. Prisms. M.p.  $127-8^\circ$  ( $125^\circ$ ). Acetyl deriv.: m.p.  $133-4^\circ$ .

Nitrile:  $\text{C}_9\text{H}_9\text{ON}$ . MW, 147. Prisms. M.p.  $58-9^\circ$ .

Me ether: see p-Methoxyhydrocinnamic Acid.

Et ether: p-ethoxyhydrocinnamic acid.  $\text{C}_{11}\text{H}_{14}\text{O}_3$ . MW, 194. Cryst. from  $\text{H}_2\text{O}$ . M.p.  $106-5^\circ$  ( $104^\circ$ ).

Carbomethoxyl: m.p.  $86-7^\circ$  ( $83-4^\circ$ ).

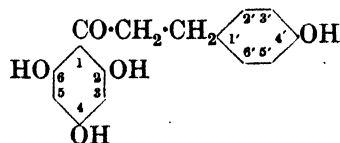
Stöhr, *Ann.*, 1884, 225, 59.

Bayer, D.R.P., 233,551, (*Chem. Zentr.*, 1911, I, 1334).

Fischer, Nouri, *Chem. Abstracts*, 1917, II, 1649.

Zemplén, Csűrös, Gerecs, Aczél, *Ber.*, 1928, 61, 2492.

**Phloretin** (Dihydronarigenin, asebogenol,  $\omega$ -p-hydroxyphenylpropylphenone)



$\text{C}_{15}\text{H}_{14}\text{O}_5$

MW, 274

Needles from  $\text{EtOH.Aq.}$  M.p.  $262-4^\circ$  ( $274^\circ$ ) decomp. (varies with rate of heating). Sol. hot  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ .

Triacetyl deriv.: m.p.  $188-9^\circ$ .

Tetra-acetyl: m.p.  $165^\circ$  ( $96^\circ$ ).

2 : 4 : 4'-Tri-Me ether:  $\text{C}_{18}\text{H}_{20}\text{O}_5$ . MW, 316. M.p.  $109-10^\circ$ . Acetyl deriv.: m.p.  $62-3^\circ$ .

2 : 6 : 4'-Tri-Me ether: m.p.  $142^\circ$ .

$\beta$ -Glucoside: see Phloridzin.

Bridel, Kramer, *Compt. rend.*, 1931, **193**, 748.

Müller, Robertson, *J. Chem. Soc.*, 1933, 1171.

Rosenmund, Rosenmund, *Ber.*, 1928, **61**, 2612.

Shinoda, Sato, Kawagoe, *Chem. Abstracts*, 1930, **24**, 604.

See also last two references above.

### Phlorhizin.

See Phloridzin.

### Phlorhizoside.

See Phloridzin.

**Phloridzin** (*Phlorrhizin*, *phlorizin*, *phlorhizin*, 2-*phloretin*- $\beta$ -glucoside, *phlorhizoside*, *asebotoside*, *asebotin*. See formula under Phloretin above)

$C_{21}H_{24}O_{10}$  MW, 436

Occurs in *Micromelum tetricarpum*, Turcz. Needles. M.p. 108°. Solidifies at 138° and remelts at 170° (about 150°).

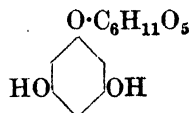
*Tri-Me ether*:  $C_{24}H_{30}O_{10}$  MW, 478. M.p. 75-6°. *Tetra-acetyl deriv.*: m.p. 94-5°.

Bridel, Kramer, *Compt. rend.*, 1931, **193**, 748.

Zemplén, Csűrös, Gerecs, Aczél, *Ber.*, 1928, **61**, 2486.

Müller, Robertson, *J. Chem. Soc.*, 1933, 1170.

### Phlorin (*Phloroglucinol*- $\beta$ -glucoside)

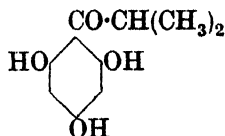


$C_{12}H_{16}O_8$  MW, 288

Cryst. M.p. 231-3°. Sol.  $H_2O$ , EtOH, MeOH. Spar. sol.  $Me_2CO$ . Insol.  $Et_2O$ , AcOEt, amyl alcohol.  $[\alpha]_D^{25} = 74.58^\circ$ .

Cremer, Seuffert, *Ber.*, 1912, **45**, 2565.

### Phlorisobutyrophenone (2 : 4 : 6-Trihydroxyisobutyrophenone)



$C_{10}H_{12}O_4$  MW, 196

Needles +  $1H_2O$  from hot  $H_2O$ . M.p. 68°, anhyd. 138-40°.

Karrer, Rosenfeld, *Helv. Chim. Acta*, 1921, **4**, 711.

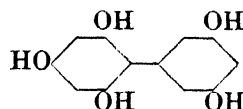
### Phlorizin.

See Phloridzin.

### Phlorobromin.

See Octabromoacetylacetone.

### Phloroglucide (*Phloroglucidol*)



Probable structure

$C_{12}H_{10}O_5$

MW, 234

Leaflets +  $2H_2O$  from  $H_2O$ . Does not melt. Spar. sol. EtOH, hot  $H_2O$ . Insol.  $Et_2O$ .

*Penta-acetyl*: m.p. 105-7°.

*Mono-Me ether*:  $C_{13}H_{12}O_5$  MW, 248.

Cryst. from  $H_2O$ . M.p. 222-5°. *Tetra-acetyl deriv.*: m.p. 102-5°.

*Penta-Me ether*:  $C_{17}H_{20}O_5$  MW, 304.

Cryst. from EtOH. M.p. 117-20°.

*Mono-Et ether*:  $C_{14}H_{16}O_5$  MW, 262.

Cryst. from  $H_2O$ . M.p. 165-8°.

Herzig, Kohn, *Monatsh.*, 1908, **29**, 677.

Wichelhaus, *Ber.*, 1919, **52**, 2054.

Cross, Bevan, *J. Chem. Soc.*, 1911, **99**, 1456.

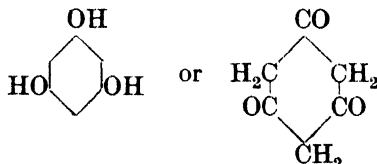
### Phloroglucidol.

See Phloroglucide.

### Phloroglucinaldehyde.

See 2 : 4 : 6-Trihydroxybenzaldehyde.

### Phloroglucinol (1 : 3 : 5-Trihydroxybenzene)



$C_6H_6O_3$

MW, 126

Leaflets or plates +  $2H_2O$  from  $H_2O$ . M.p. 117°, 217-19° anhyd., rapid heat.; 200-9° slow heat. Sol. EtOH,  $Et_2O$ , Py. Mod. sol.  $H_2O$ . Heat of comb.  $C_v$  617.65 Cal.  $k = 4.5 \times 10^{-10}$  at 25°.  $FeCl_3$  in EtOH  $\rightarrow$  bluish-violet col. Sol. in  $H_2O$  + drop  $NH_3$ . Aq.  $\rightarrow$  violet-red col.

$C_6H_6O_3 \cdot 1NH_3$ : m.p. 88-91°.

$2C_6H_6O_3 \cdot (C_2H_5)_3N$ : m.p. 103-4°.

*Triacetyl*: m.p. 104°.

*Tribenzoyl*: m.p. 185°.

*Mono-p-hydroxybenzoyl deriv.*: m.p. 218°.

*Trioxime*: blackens at 140°. Explodes at 155°.

*Picrate*: m.p. 101-3°.



*Mono-Me ether*:  $C_7H_8O_3$ . MW, 140. M.p.  $78^\circ$  ( $78-81^\circ$ ,  $77-80^\circ$ ). B.p.  $213^\circ/16$  mm.,  $188-9^\circ/12$  mm.

*Di-Me ether*:  $C_8H_{10}O_3$ . MW, 154. Cryst. from  $C_6H_6$ -ligroin. M.p.  $36-8^\circ$ . B.p.  $172-5^\circ/17$  mm.

*Tri-Me ether*:  $C_9H_{12}O_3$ . MW, 168. Prisms from EtOH. M.p.  $52.5^\circ$  ( $54-5^\circ$ ). B.p.  $255.5^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Mono-Et ether*:  $C_8H_{10}O_3$ . MW, 154. Leaflets +  $2H_2O$  from  $H_2O$ . M.p.  $84-6^\circ$ . B.p.  $220^\circ/15$  mm. ( $220-1^\circ/30$  mm.).

*Di-Et ether*:  $C_{10}H_{14}O_3$ . MW, 182. Needles from  $H_2O$ . M.p.  $88-9^\circ$ . B.p.  $188-9^\circ/20$  mm. Volatile in steam. *Me ether*:  $C_{11}H_{16}O_3$ . MW, 196. B.p.  $147-8^\circ/13$  mm.

*Tri-Et ether*:  $C_{12}H_{18}O_3$ . MW, 210. Cryst. M.p.  $43^\circ$ . B.p.  $175^\circ/24$  mm. Sol. EtOH,  $Et_2O$ . Volatile in steam.

*Triphenyl ether*:  $C_{24}H_{18}O_3$ . MW, 354. Prisms from  $Et_2O$ . M.p.  $112^\circ$ . B.p.  $290-3^\circ/20$  mm. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. EtOH, AcOH, ligroin.

*Dibenzyl ether*:  $C_{20}H_{18}O_3$ . MW, 306. M.p.  $62-4^\circ$ . Sol. ord. org. solvents.

*Tribenzyl ether*:  $C_{27}H_{24}O_3$ . MW, 396. M.p.  $39-41^\circ$ . Sol. ord. org. solvents.

*Glucoside*: see Phlorin.

Herzig, Aigner, *Monatsh.*, 1900, **21**, 444.

Clarke, Hartman, *Organic Syntheses*, 1929, IX, 74.

Goris, Canal, *Compt. rend.*, 1935, **201**, 1435.

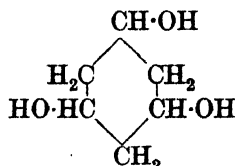
### Phloroglucinol-dicarboxylic Acid.

See 2 : 4 : 6-Trihydroxyisophthalic Acid.

### Phloroglucite.

See Phloroglucitol.

**Phloroglucitol** (*Phloroglucite*, hexahydro-phloroglucinol, 1 : 3 : 5-trihydroxycyclohexane, cyclohexantriol-1 : 3 : 5)



$C_6H_{12}O_3$

MW, 132

( $\alpha$ ) Cryst. +  $2H_2O$  from  $H_2O$ . M.p. about  $110^\circ$  decomp.  $\rightarrow$  solid remelting at  $184^\circ$ . Spar. sol.  $C_6H_6$ ,  $Me_2CO$ .

*Triacetyl*: m.p.  $79^\circ$ .

*Tri-phenylurethane*: m.p.  $245^\circ$ .

( $\beta$ ) Cryst. from EtOH. M.p.  $145^\circ$ . Sol.  $H_2O$ . Spar. sol.  $C_6H_6$ ,  $Me_2CO$ .

*Tri-phenylurethane*: m.p.  $160^\circ$ .

Lindemann, Baumann, *Ann.*, 1930, **477**, 78.

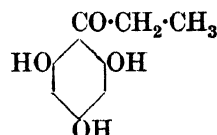
### Phlorol.

*o*-Ethylphenol, *q.v.*

### Phlorone.

See *p*-Xylo-*p*-quinone.

**Phlorpropiophenone** (2 : 4 : 6-Trihydroxy-propioophenone)



$C_9H_{10}O_4$

MW, 182

Needles +  $1H_2O$  from  $H_2O$ . M.p.  $174-5^\circ$ . Sol. EtOH, warm  $H_2O$ . Alc.  $FeCl_3 \rightarrow$  purple col.

2 : 4-Di-Me ether:  $C_{11}H_{14}O_4$ . MW, 210. Plates from EtOH. M.p.  $111^\circ$ .  $FeCl_3 \rightarrow$  wine-red col.

2 : 6-Di-Me ether: prisms from AcOEt. M.p.  $180^\circ$ . No col. with  $FeCl_3$ . *Acetyl*: plates from EtOH.Aq. M.p.  $76^\circ$ . *Benzoyl*: needles from MeOH. M.p.  $103^\circ$ .

2-Benzoyl: prisms from MeOH. M.p.  $191-2^\circ$ . Alc.  $FeCl_3 \rightarrow$  wine-red col.

4-Benzoyl: needles from MeOH. M.p.  $193^\circ$ . Alc.  $FeCl_3 \rightarrow$  reddish-brown col.

Canter, Curd, Robertson, *J. Chem. Soc.*, 1931, 1245.

Howells, Little, *J. Am. Chem. Soc.*, 1932, **54**, 2452.

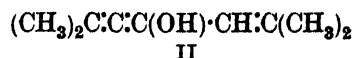
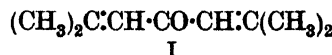
### Phlorrhizin.

See Phloridzin.

### Phonopyrrole.

Hæmopyrrole, *q.v.*

**Phorone** (2 : 6-Dimethyl-2 : 5-heptadienone-4, di-isopropylideneacetone)



$C_9H_{14}O$

MW, 138

I.

Yellowish-green prisms. M.p.  $28^\circ$ . B.p.  $197.2^\circ/743.3$  mm.  $D_4^{20}$  0.8850.  $n_D^{20}$  1.49982.

*Oxime*: m.p.  $48^\circ$ . B.p.  $218^\circ$ .

## II.

(a) B.p. 79.8°/14 mm.  $D_4^{18.1}$  0.8854.  $n_D^{18.1}$  1.48847. (b) B.p. 81.8–82°/14 mm.  $D_4^{18}$  0.8893.  $n_D^{18}$  1.4932.

Auwers, Eisenlohr, *J. prakt. Chem.*, 1911, **84**, 76.

Francis, *J. Chem. Soc.*, 1927, 2898.

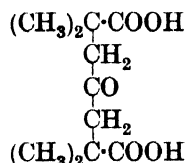
I.G., D.R.P., 483,823, (*Chem. Abstracts*, 1930, **24**, 2146).

Sugden, *J. Chem. Soc.*, 1928, 412.

Pereira, *Chem. Abstracts*, 1910, **4**, 2275.

Claissen, *Ann.*, 1876, **180**, 4.

**Phoronic Acid** (3-Keto-1 : 1 : 5 : 5-tetramethyl-pimelic acid)



$\text{C}_{11}\text{H}_{18}\text{O}_5$  MW, 230

Prisms from EtOH.Aq. M.p. 184° decomp. Sol. EtOH. Spar. sol. hot  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_{12}\text{H}_{20}\text{O}_5$ . MW, 244. Prisms from MeOH. M.p. 105–6°.

*Di-Me ester*:  $\text{C}_{13}\text{H}_{22}\text{O}_5$ . MW, 258. Prisms from pet. ether. M.p. 32°.

*Di-Et ester*:  $\text{C}_{15}\text{H}_{26}\text{O}_5$ . MW, 286. M.p. 33° (125°).

*Di-lactone (anhydride)*:  $\text{C}_{11}\text{H}_{16}\text{O}_4$ . MW, 212. M.p. 138° (132°).

Toivonen, *Chem. Zentr.*, 1928, II, 39.

Milikan, *Rec. trav. chim.*, 1912, **31**, 287.

**Phosgene.**

See Carbonyl chloride.

**Phosphaniline.**

See Phenylphosphine.

**Phosphinobenzene.**

See under Phenylphosphorous Acid.

**Phosphobenzene (Diphosphenyl)**

$\text{C}_{12}\text{H}_{10}\text{P}_2$  MW, 216

Yellow powder. M.p. 149–50°. Sol. hot  $\text{C}_6\text{H}_6$ . Insol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ .

Köhler, Michaelis, *Ber.*, 1877, **10**, 812.

**Phosphoric Acid. Organic derivatives.**

*Tri-Me ester*:  $\text{C}_3\text{H}_9\text{O}_4\text{P}$ . MW, 140. B.p. 197°, 97°/36 mm., 85°/24 mm.  $D^{22}$  1.200.

*Tri-Et ester*:  $\text{C}_6\text{H}_{15}\text{O}_4\text{P}$ . MW, 182. B.p. 215°, 190°/445 mm., 161°/188 mm., 146°/112 mm., 123°/50 mm., 103°/25 mm., 98–98.5°/8–10 mm.  $D_0^{19}$  1.0725.  $n_D^{17.1}$  1.40674.

*Tripropyl ester*:  $\text{C}_9\text{H}_{21}\text{O}_4\text{P}$ . MW, 224. B.p. 138°/47 mm., 133.5°/22 mm., 120.5–121.5°/8–10 mm.  $D^{22}$  1.007.

*Tri-isopropyl ester*: b.p. 218–20°/763 mm., 136°/68 mm., 95–6°/8–10 mm.

*Tributyl ester*:  $\text{C}_{12}\text{H}_{27}\text{O}_4$ . MW, 266. B.p. 160–2°/15 mm.

*Tri-isobutyl ester*: b.p. 192°, 119–29°/8–12 mm.  $D^{22}$  0.965.

*Tri-n-amyl ester*:  $\text{C}_{15}\text{H}_{33}\text{O}_4\text{P}$ . MW, 308. B.p. 158–63°/6 mm.

*Monohexadecyl ester*:  $\text{C}_{16}\text{H}_{35}\text{O}_4\text{P}$ . MW, 322. M.p. 74°.

*Triallyl ester*:  $\text{C}_9\text{H}_{15}\text{O}_4\text{P}$ . MW, 218. B.p. 157°/44 mm.

*Phenyl ester*: see Diphenyl phosphate, Triphenyl phosphate, and under Phenol.

*Naphthyl ester*: see under Naphthol.

*Dibenzyl ester*:  $\text{C}_{14}\text{H}_{15}\text{O}_4\text{P}$ . MW, 278. M.p. 78–9°.

*Tribenzyl ester*:  $\text{C}_{21}\text{H}_{21}\text{O}_4\text{P}$ . MW, 368. M.p. 64°.

*Dianilide*: m.p. 214–16°.

*Trianilide*: m.p. 212–13° (208–10°).

*Di-o-toluidide*: m.p. 120°.

*Di-p-toluidide*: m.p. 195°.

*Tri-o-toluidide*: m.p. 236°.

*Tri-p-toluidide*: m.p. 192–4°.

*Tri-benzylamide*: m.p. 98°.

*Tri-1-naphthylamide*: m.p. 216°.

*Tri-2-naphthylamide*: m.p. 170°.

Adler, Gottlieb, U.S.P., 1,983,588, (*Chem. Abstracts*, 1935, **29**, 817).

Harlay, *Chem. Abstracts*, 1935, **29**, 2506.

Chem. Fabrik v. Heyden, E.P., 398,659, (*Chem. Abstracts*, 1934, **28**, 1362).

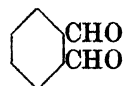
Noller, Dutton, *J. Am. Chem. Soc.*, 1933, **55**, 424.

**Phosphorus triethyl.**

See Triethylphosphine.

**Phrenosin.**

See Cerebron.

**o-Phthalaldehyde (1 : 2-Dialdehydobenzene)**

$\text{C}_8\text{H}_6\text{O}_2$

MW, 134

(1) Cryst. from ligroin. M.p. 53.2°.

(2) Yellow cryst. M.p. 56–7°.

Sol.  $\text{H}_2\text{O}$ , ord. org. solvents. Spar. sol. pet. ether. Volatile in steam.  $\text{CrO}_3 \rightarrow$  phthalic acid.

*Di-phenylhydrazone*: m.p. 190–1°.

*Tetra-acetate* : m.p. 126–7°.

Thiele, Günther, *Ann.*, 1906, **347**, 107.

Seekles, *Rec. trav. chim.*, 1923, **42**, 706.

Bayer, D.R.P., 121,788, (*Chem. Zentr.*, 1901, II, 70).

**m-Phthalaldehyde.**

See Isophthalaldehyde.

**p-Phthalaldehyde.**

See Terephthalaldehyde.

**o-Phthalaldehydic Acid.**

See o-Aldehydobenzoic Acid.

**Phthalamic Acid** (*Phthalic acid monoamide, o-carbamylbenzoic acid*)



$C_8H_7O_3N$

MW, 165

Prisms. M.p. 148–9°. Mod. sol. EtOH, hot  $H_2O$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Insol. ligroin. Heat of comb.  $C_p$  850.7 Cal.  $k = 1.6 \times 10^{-4}$  at 25°. Heat at 155°  $\rightarrow$  phthalimide.

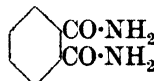
2 : 4-Dinitrophenylhydrazones : m.p. 298–9°.

Fodor, F.P., 636,846, (*Chem. Abstracts*, 1929, **23**, 608).

Chesnais, Canadian P., 282,407, (*Chem. Abstracts*, 1928, **22**, 4133).

Aschan, *Ber.*, 1886, **19**, 1402.

**Phthalamide** (*Phthalic acid diamide*)



$C_8H_8O_2N_2$

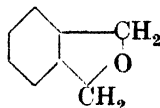
MW, 164

Cryst. M.p. 219–20° slow heat.  $\rightarrow$  phthalimide +  $NH_3$ . Spar. sol.  $H_2O$ , EtOH. Heat of comb.  $C_p$  291.7 Cal.

Hoogewerff, van Dorp, *Rec. trav. chim.*, 1892, **11**, 100.

Aschan, *Ber.*, 1886, **19**, 1399.

**Phthalan** (o-Xylylene oxide, 2 : 5-dihydro-3 : 4-benzofuran, isocoumaran)



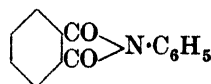
$C_8H_8O$

MW, 120

Oil with odour of benzaldehyde. B.p. 192°. Volatile in steam.  $D_4^{20}$  1.098. Alk.  $KMnO_4 \rightarrow$  phthalic acid.

Ludwig, *Ber.*, 1907, **40**, 3062.

**Phthalanil** (*N-Phenylphthalimide*)



$C_{14}H_9O_2N$

MW, 223

Needles from EtOH. M.p. 210° (208°, 206°, 203°). Sol.  $CHCl_3$ . Insol.  $H_2O$ . Sublimes.

Cornillot, *Ann. chim.*, 1927, **8**, 177.

Jolles, *Gazz. chim. ital.*, 1935, **65**, 1224.

Warren, Briggs, *Ber.*, 1931, **64**, 29.

Das, Sarker, *J. Indian Chem. Soc.*, 1934, **11**, 709.

Porai-Koshitz, *Chem. Abstracts*, 1935, **29**, 131, 6590.

See also Sherrill, Schaeffer, Shoyer, *J. Am. Chem. Soc.*, 1928, **50**, 477.

**Phthalanilic Acid** (*Phthalic acid monoanilide*)



$C_{14}H_{11}O_3N$

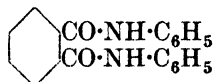
MW, 241

Needles from EtOH. M.p. 170°  $\rightarrow$  phthanal.

Anhydride : see Phthalanil.

See last four references above.

**Phthalanilide** (*N : N'-Diphenylphthalamide, phthalic acid dianilide*)



$C_{20}H_{16}O_2N_2$

MW, 316

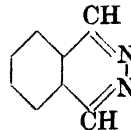
Needles from EtOH. M.p. 251° (245–50°, 231° decomp.). Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Heat of comb.  $C_p$  2383.2 Cal.

Tingle, Cram, *Am. Chem. J.*, 1907, **37**, 603.

**Phthalanone.**

See Phthalide.

**Phthalazine** (4 : 5-Benzodiazine,  $\beta$ -phenodiazine)



$C_8H_6N_2$

MW, 130

Needles from  $Et_2O$ . M.p. 90–1°. B.p. about 315–17° decomp., 189°/29 mm., 175°/17 mm. Sol.  $H_2O$ , EtOH,  $C_6H_6$ . Spar. sol.  $Et_2O$ . Insol. ligroin.

$B, HCl$  : m.p. 231° decomp.

$B, HI$  : m.p. 203°.

$B, HAuCl_4$  : m.p. 200°.

*Methiodide* : m.p. 235–40°.

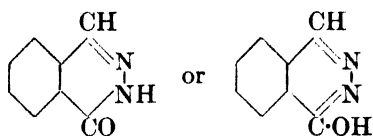
*Ethiodide* : m.p. 204–10°.

*Benzylchloride* : m.p. 97–9°.

*Picrate* : m.p. 208–10°.

Paul, *Ber.*, 1899, 32, 2015.

### Phthalazone



$C_8H_6ON_2$  MW, 146

Needles from  $H_2O$ . M.p. 183–4°. B.p. 337°. Sol.  $H_2O$ , EtOH,  $C_6H_6$ . Sublimes in prisms.

*N-Acetyl* : m.p. 135°.

*N-Me* :  $C_9H_8ON_2$ . MW, 160. M.p. 114° (111–12°).

*O-Me* : needles. M.p. 60–1°.

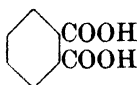
*N-Et* :  $C_{10}H_{10}ON_2$ . MW, 174. M.p. 55° (67–8°). B.p. 295°.

*O-Et* : m.p. 29–31°.

Rothenburg, *J. prakt. Chem.*, 1895, 51, 147.

Paul, *Ber.*, 1899, 32, 2020.

### Phthalic Acid (*Benzene-o-dicarboxylic acid*)



$C_8H_6O_4$  MW, 166

Plates from hot  $H_2O$ . M.p. 231° (rapid heat.), 191° (in sealed tube). Mod. sol.  $H_2O$ , EtOH. Spar. sol. Et<sub>2</sub>O. Insol.  $CHCl_3$ . Heat of comb.  $C_p$  771.6 Cal.,  $C_v$  779.3 Cal.  $k$  (first) =  $1.3 \times 10^{-3}$  at 25°; (second) =  $3.9 \times 10^{-6}$  at 18°.

*Aniline salt* : m.p. 158°.

*Pyridine acid salt* : m.p. 86°.

*Me ester* :  $C_9H_8O_4$ . MW, 180. M.p. 85° (82–5°).  $k = 6.56 \times 10^{-4}$  at 25°.

*Di-Me ester* :  $C_{10}H_{10}O_4$ . MW, 194. B.p. 282°.  $D_4^{20.7}$  1.1905.  $n_D^{20.7}$  1.515. Heat of comb.  $C_p$  1120.4 Cal.,  $C_v$  1120.1 Cal.

*Me-Et ester* :  $C_{11}H_{12}O_4$ . MW, 208. B.p. 285–7° (281–2°).

*Et ester* :  $C_{10}H_{10}O_4$ . MW, 194. M.p. 2°.  $D_4^{23}$  1.1877.  $n_D^{23}$  1.509.  $k = 5.51 \times 10^{-4}$  at 25°.

*Di-Et ester* : see Diethyl phthalate.

*Dibutyl ester* :  $C_{18}H_{22}O_4$ . MW, 278. B.p. 212–15°.  $D_4^{21}$  1.043–1.050.

*d-Isobutyl ester* :  $C_{12}H_{14}O_4$ . MW, 222. M.p. 46–7°.  $[\alpha]_D^{20} + 38.4^\circ$  in EtOH.

*dl-Isobutyl ester* : cryst. from pet. ether. M.p. 56–7°.

*Di-isobutyl ester* : b.p. 182–4°/10 mm.

*Dihexadecyl ester* :  $C_{40}H_{70}O_4$ . MW, 614. M.p. 43°.

*Cyclohexyl ester* :  $C_{14}H_{16}O_4$ . MW, 248. M.p. 99°.

*Di-cyclohexyl ester* :  $C_{20}H_{26}O_4$ . MW, 330. M.p. 66°.

*Phenyl ester* :  $C_{14}H_{10}O_4$ . MW, 242. Needles from  $C_6H_6$ –ligroin. M.p. 103°.

*Diphenyl ester* :  $C_{20}H_{14}O_4$ . MW, 318. Prisms from EtOH. M.p. 73° (70°). B.p. 250–7°/14 mm.

*Di-1-naphthyl ester* :  $C_{28}H_{18}O_4$ . MW, 418. M.p. 155°.

*Benzyl ester* :  $C_{15}H_{12}O_4$ . MW, 256. M.p. 106–7°.

*Dibenzyl ester* :  $C_{22}H_{18}O_4$ . MW, 346. M.p. 42–3°. B.p. 277°/15 mm., 274°/12 mm.

*Di-p-nitrobenzyl ester* : m.p. 154–5°.

*Di-fluoride* : see Phthaloyl fluoride.

*Dichloride* : see Phthaloyl chloride.

*Monoamide* : see Phthalamic Acid.

*Diamide* : see Phthalamide.

*Mononitrile* : see o-Cyanobenzoic Acid.

*Dinitrile* : see Phthalonitrile.

*Monoanilide* : see Phthalanilic Acid.

*Dianilide* : see Phthalanilide.

*Anhydride* : see Phthalic Anhydride.

*Imide* : see Phthalimide.

*Mono-phenylhydrazide* : m.p. 165–6°.

*Di-phenylhydrazide* : m.p. 161°.

Pickard, Kenyon, *J. Chem. Soc.*, 1913, 103, 1937; 1914, 105, 1126.

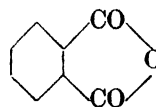
Miller, *Chem. Zentr.*, 1914, I, 790.

Buchsweiler, D.R.P., 232,818, (*Chem. Zentr.*, 1911, I, 1090).

Reid, *J. Am. Chem. Soc.*, 1917, 39, 1249.

Dennstedt, Hassler, D.R.P., 203,848, (*Chem. Zentr.*, 1908, II, 1750).

### Phthalic Anhydride



$C_8H_4O_3$

MW, 148

Needles from EtOH or  $C_6H_6$ . M.p. 131–61°.

B.p. 295.09° (285.1°), 284.54°/751.4 mm., 283.75°/739 mm. Sol. EtOH. Spar. sol. Et<sub>2</sub>O,  $H_2O$ . Sublimes. Sp. gr. 1.527. Intermediate for dyestuffs of the pyronine or phthalein class (Fluorescein, Rhodamine, Eosine, Phloxine, etc.) and for synthesis of anthraquinones, glyptal resins, and many other industrial products.

*Oxime* : see Phthaloxime.

*Mono-2-pyridylhydrazide*: m.p. 208°.

*Mono-2-quinolylhydrazide*: m.p. 236°.

Punnett, U.S.P., 1,978,506, (*Chem. Abstracts*, 1935, **29**, 176).

National Aniline, E.P., 415,748, (*Chem. Abstracts*, 1935, **29**, 819); U.S.P., 2,004,586, (*ibid.*, 5128).

Douglass, Jones, U.S.P., 2,013,727, (*Chem. Abstracts*, 1935, **29**, 6903).

Buylla, Pertierra, *Chem. Abstracts*, 1933, **27**, 1624.

Timmermans, Burrul, *Chimie et industrie*, 1931, Suppl. No., 196.

Marti, *Bull. soc. chim. Belg.*, 1930, **39**, 590.

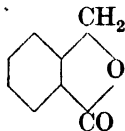
Andrews, *Ind. Eng. Chem.*, 1921, **13**, 167.

Gibbs, *Ind. Eng. Chem.*, 1920, **12**, 1017 (*Bibl.*).

### Phthalidanil.

See *N*-Phenylphthalimidine.

**Phthalide** (1-*Phthalanone*, *o*-hydroxymethylbenzoic acid lactone, 1(2)-isobenzfuranone, 3:4-benzfuranone-2)



$C_8H_6O_2$

MW, 134

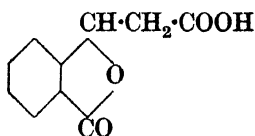
(i) Needles or plates from  $H_2O$ . M.p. 75° (73-4°). Stable. (ii) M.p. 65-8° (65°). Labile. Sol. EtOH, Et<sub>2</sub>O, hot  $H_2O$ .  $D_4^{20}$  1.1636.  $n_D^{20}$  1.536. Heat of comb.  $C_p$  885.1 Cal.,  $C_v$  884.7 Cal.

Jaeger, U.S.P., 1,889,961, (*Chem. Abstracts*, 1933, **27**, 1640).

Kalle, D.R.P., 267,596, (*Chem. Zentr.*, 1914, I, 199).

Gardner, Naylor, *Organic Syntheses*, 1936, XVI, 71.

### Phthalide-3-acetic Acid



$C_{10}H_8O_4$

MW, 192

Cryst. +  $1H_2O$  from  $H_2O$ . M.p. 151.5°. Sol. EtOH, hot  $H_2O$ .

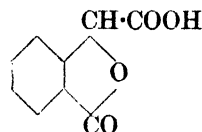
*Me ester*:  $C_{11}H_{10}O_4$ . MW, 206. M.p. 62°.

*Et ester*:  $C_{12}H_{12}O_4$ . MW, 220. M.p. 76°.

Gabriel, Michael, *Ber.*, 1877, **10**, 1558.

Roth, *Ber.*, 1914, **47**, 1598.

### Phthalide-3-carboxylic Acid



$C_9H_6O_4$

MW, 178

Leaflets from  $C_6H_6$ -AcOH. M.p. 153° (151°). Sol.  $H_2O$ , EtOH, Et<sub>2</sub>O, AcOH.

*Me ester*:  $C_{10}H_8O_4$ . MW, 192. Needles from ligroin. M.p. 57° (53-4°).

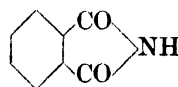
*Amide*:  $C_9H_7O_3N$ . MW, 177. Cryst. from  $H_2O$ . M.p. 185-5°.

Fries, *Ann.*, 1904, **334**, 358.

Tscherniac, *J. Chem. Soc.*, 1916, **109**, 1240.

Ruhemann, *J. Chem. Soc.*, 1910, **97**, 2030.

### Phthalimide



$C_8H_5O_2N$

MW, 147

Needles from  $H_2O$ , prisms from AcOH, leaflets by sublimation. M.p. 233-5° (238°). Sol. hot AcOH. Insol.  $C_6H_6$ , ligroin. Forms *N*-metallic derivs.

*N-Acetyl*: m.p. 133-5°.

*N-Chloro*: chlorophthalimide.  $C_8H_4O_2NCl$ . MW, 181.5. M.p. 183-5°.

*N-Bromo*:  $C_8H_4O_2NBr$ . MW, 226. Yellow cryst. from  $C_6H_6$ . M.p. 206-7°.

*N-Me*: see *N*-Methylphthalimide.

*N-Et*:  $C_{10}H_9O_2N$ . MW, 175. Needles from EtOH. M.p. 78-9°. B.p. 285°/758 mm.

*N-2-Chloroethyl*:  $C_{10}H_8O_2NCl$ . MW, 209.5. M.p. 79-81°.

*N-2-Bromoethyl*:  $C_{10}H_8O_2NBr$ . MW, 254. M.p. 82-83.5°.

*N-2-Iodoethyl*:  $C_{10}H_8O_2NI$ . MW, 301. M.p. 99-100°.

*N-Propyl*:  $C_{11}H_{11}O_2N$ . MW, 189. M.p. 66°. B.p. 296-9°/758 mm. (282-3°/756 mm.).

*N-2-Chloropropyl*:  $C_{11}H_{10}O_2NCl$ . MW, 223.5. M.p. 100-2°.

*N-3-Chloropropyl*: m.p. 67-8°.

*N-2:3-Dichloropropyl*:  $C_{11}H_9O_2NCl_2$ . MW, 258. M.p. 93°.

*N-2-Bromopropyl*:  $C_{11}H_{10}O_2NBr$ . MW, 268. M.p. 110-11° (105°).

*N-3-Bromopropyl*: m.p. 72-3°.

N-2 : 3-Dibromopropyl :  $C_{11}H_9O_2NBr_2$ . MW, 347. M.p. 113–14°.

N-3-Iodopropyl :  $C_{11}H_{10}O_2NI$ . MW, 315. M.p. 88°.

N-Isopropyl : m.p. 85°. B.p. 286°/761 mm.

N-2-Chloroisopropyl : m.p. 56–8°.

N-2-Bromoisopropyl : m.p. 59–60°.

N-Butyl :  $C_{12}H_{13}O_2N$ . MW, 203. B.p. 311–18°/758 mm.

N-4-Bromobutyl :  $C_{12}H_{12}O_2NBr$ . MW, 282. M.p. 80–5°.

N-4-Iodobutyl :  $C_{12}H_{12}O_2NI$ . MW, 329. M.p. 88–89–5°.

N-Isobutyl : m.p. 93°. B.p. 293–5°.

N-Amyl :  $C_{13}H_{15}O_2N$ . MW, 217. M.p. 23°. B.p. 303°.  $D_4^{25}$  1.093.  $[\alpha]_D^{25} + 7.53^\circ$ .

N-Isoamyl : m.p. 12–5°. B.p. 307–8°.

N-Octyl :  $C_{16}H_{21}O_2N$ . MW, 259. M.p. 48–9°. B.p. about 216°/20 mm.

N-Cyclopropyl :  $C_{11}H_9O_2N$ . MW, 187. M.p. 135–6°.

N- $\alpha$ -Camphyl :  $C_{18}H_{21}O_2N$ . MW, 283. M.p. 54°.

N-Vinyl :  $C_{10}H_7O_2N$ . MW, 173. M.p. 86°.

N-Propenyl :  $C_{11}H_9O_2N$ . MW, 187. Yellow leaflets from EtOH. M.p. 151°.

N-Isopropenyl : m.p. 105–6°.

N-Allyl :  $C_{11}H_9O_2N$ . MW, 187. Cryst. from EtOH. M.p. 71°. B.p. 295°.

N-Phenyl : see Phthalanil.

N-p-Chlorophenyl :  $C_{14}H_8O_2NCl$ . MW, 257.5. M.p. 194–5°.

N-2 : 4-Dichlorophenyl :  $C_{14}H_7O_2NCl_2$ . MW, 292. M.p. 155°.

N-p-Bromophenyl :  $C_{14}H_8O_2NBr$ . MW, 302. M.p. 203–4°.

N-2 : 4-Dibromophenyl :  $C_{14}H_7O_2NBr_2$ . MW, 381. M.p. 153–5°.

N-p-Iodophenyl :  $C_{14}H_8O_2NI$ . MW, 349. M.p. 227–8° (235°).

N-o-Nitrophenyl :  $C_{14}H_8O_4N_2$ . MW, 268. M.p. 202–3°.

N-m-Nitrophenyl : m.p. 242–4°.

N-p-Nitrophenyl : m.p. 264–6°.

N-Picryl :  $C_{14}H_6O_8N_4$ . MW, 358. M.p. 259°.

N-Benzyl :  $C_{15}H_{11}O_2N$ . MW, 237. M.p. 115–16°.

N-o-Tolyl :  $C_{15}H_{11}O_2N$ . MW, 237. M.p. 182°.

N-m-Tolyl : m.p. 170–2°.

N-p-Tolyl : m.p. 204°.

N-Cinnamyl :  $C_{17}H_{13}O_2N$ . MW, 263. M.p. 153°.

N-1-Naphthyl :  $C_{18}H_{11}O_2N$ . MW, 273. M.p. 180–1°.

N-2-Naphthyl : m.p. 216°.

Herzog, *Z. angew. Chem.*, 1919, **32**, 301.

Tseng, *Chem. Abstracts*, 1934, **28**, 6133.

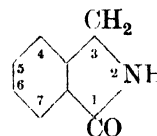
Inone, Horiguchi, *Chem. Abstracts*, 1933, **27**, 2684.

Jaeger, Daniels, U.S.P., 1,966,068, (*Chem. Abstracts*, 1934, **28**, 5476); U.S.P., 1,914,723, (*Chem. Abstracts*, 1933, **27**, 4243); U.S.P., 1,968,253, (*Chem. Abstracts*, 1934, **28**, 5835).

Manske, *Organic Syntheses*, 1932, XII, 10.

Salzberg, Supniewski, *Organic Syntheses*, 1927, VII, 8.

**Phthalimidine** (1-Isoindolenone, benzylamine-o-carboxylic lactam)



$C_8H_7ON$  MW, 133

Needles from  $H_2O$ . M.p. 150° (149°). B.p. 336–7°/730 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

B, HCl : m.p. 150° decomp.

N-Acetyl : m.p. 151°.

N-Me :  $C_9H_9ON$ . MW, 147. M.p. 120°. B.p. 300°.

N-Et :  $C_{10}H_{11}ON$ . MW, 161. M.p. 45°.  $B_2, H_2PtCl_6$  : m.p. 145°.

N-Isobutyl :  $C_{12}H_{15}ON$ . MW, 189. B.p. 310–12°/740 mm.

N-Phenyl : see N-Phenylphthalimidine.

N-Benzyl :  $C_{15}H_{13}ON$ . MW, 223. M.p. 90–1°.

N-o-Hydroxybenzyl :  $C_{15}H_{13}O_2N$ . MW, 239. M.p. 159–60°.

N-p-Hydroxybenzyl : red needles from  $H_2O$ . M.p. 187–98°.

N-o-Aminobenzyl :  $C_{15}H_{14}ON_2$ . MW, 238. M.p. 153–4°.

N-p-Aminobenzyl : m.p. 187–8°.

N-Nitroso : m.p. 150°.

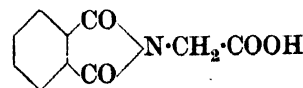
Picrate : m.p. 140°.

Rupe, Bernstein, *Helv. Chim. Acta*, 1930, **13**, 469.

Sakurai, *Bull. Chem. Soc. Japan*, 1930, **5**, 184.

Packendorff, *Ber.*, 1934, **67**, 907.

**Phthaliminoacetic Acid** (Phthaloylglycine)



$C_{10}H_7O_4N$

MW, 205

Prisms or needles. From  $\text{H}_2\text{O}$ . M.p.  $192-3^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , hot  $\text{EtOH}$ . Insol.  $\text{CHCl}_3$ , pet. ether, ligroin.  $k = 1 \times 10^{-3}$  at  $25^\circ$ .

*Et ester*:  $\text{C}_{12}\text{H}_{11}\text{O}_4\text{N}$ . MW, 233. Needles from  $\text{H}_2\text{O}$ . M.p.  $104-5^\circ$  ( $112-13^\circ$ ).

*Chloride*:  $\text{C}_{10}\text{H}_5\text{O}_3\text{NCl}$ . MW, 222.5. Needles from ligroin. M.p.  $84-5^\circ$ .

*Anhydride*:  $\text{C}_{20}\text{H}_{12}\text{O}_7\text{N}_2$ . MW, 392. Needles from  $\text{PhNO}_2$ . M.p.  $242^\circ$ .

*Anilide*: m.p.  $231-2^\circ$ .

*Phenylhydrazide*: m.p.  $199^\circ$ .

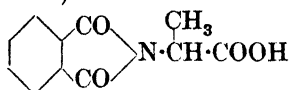
Johnson, Scott, *J. Am. Chem. Soc.*, 1913, 35, 1133.

Reise, *Ann.*, 1887, 242, 1.

Gabriel, *Ber.*, 1907, 40, 2649.

Scheiber, *Ber.*, 1913, 46, 1103.

### 1-Phthaliminopropionic Acid (N-Phthaloylalanine)



$\text{C}_{11}\text{H}_9\text{O}_4\text{N}$  MW, 219  
d-.

*Et ester*:  $\text{C}_{13}\text{H}_{13}\text{O}_4\text{N}$ . MW, 247. Cryst. from ligroin. M.p.  $58-61^\circ$ .

l-.

M.p.  $150-1^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ . Spar. sol. ligroin.  $[\alpha]_D^{20} = -17.8^\circ$  in  $\text{EtOH}$ .

*Et ester*: cryst. from ligroin. M.p.  $54-6^\circ$ .  $[\alpha]_D^{20} = -12.5^\circ$  in  $\text{EtOH}$ .

dl-.

Needles from  $\text{H}_2\text{O}$ . M.p.  $160-2^\circ$  ( $164^\circ$ ). Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{AcOH}$ , hot  $\text{H}_2\text{O}$ . Insol. pet. ether.

*Et ester*: plates from  $\text{CS}_2$ . M.p.  $65^\circ$  ( $61-3^\circ$ ).

*Phenyl ester*:  $\text{C}_{17}\text{H}_{13}\text{O}_4\text{N}$ . MW, 295. Needles from  $\text{EtOH}$ . M.p.  $99^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{AcOH}$ , hot  $\text{EtOH}$ .

*Chloride*:  $\text{C}_{11}\text{H}_8\text{O}_3\text{NCl}$ . MW, 237.5. Cryst. from ligroin. M.p.  $73^\circ$  ( $71^\circ$ ).

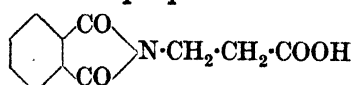
Bayer, D.R.P., 209,962, (*Chem. Zentr.*, 1909, I, 1951).

Bachstetz, *Ber.*, 1914, 47, 3166.

Fischer, *Ber.*, 1907, 40, 498.

Gabriel, *Ber.*, 1905, 38, 634.

### 2-Phthaliminopropionic Acid



$\text{C}_{11}\text{H}_9\text{O}_4\text{N}$  MW, 219

Cryst. from  $\text{H}_2\text{O}$ . M.p.  $150-1^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{AcOH}$ ,  $\text{AcOEt}$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ . Insol. ligroin.

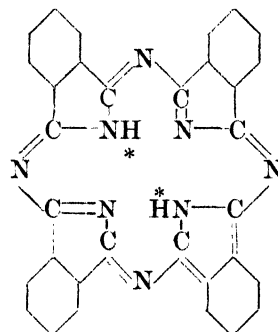
*Et ester*: needles. M.p.  $73.5^\circ$ .

*Isoamyl ester*:  $\text{C}_{16}\text{H}_{19}\text{O}_4\text{N}$ . MW, 289. M.p.  $61^\circ$ .

*Chloride*: m.p.  $107-8^\circ$ .

Hale, Britton, *J. Am. Chem. Soc.*, 1919, 41, 845.

### Phthalocyanine



$\text{C}_{32}\text{H}_{18}\text{N}_8$  MW, 514

Greenish-blue needles with purple reflex from quinoline. Sublimes at  $550^\circ$  decomp. under red. press. Sol. benzophenone, naphthalene, cyclohexanol, menthol, quinoline  $\rightarrow$  blue sols. Sol. aniline and homologues  $\rightarrow$  green sols. Very stable. Forms metallic derivs. at N atoms marked \*, of formula  $(\text{C}_8\text{H}_4\text{N}_2)_4\text{M}$ , very stable blue cryst. solids.

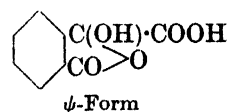
Robertson, *J. Chem. Soc.*, 1935, 615.

Linstead, *J. Chem. Soc.*, 1934, 1020, 1035.

Barnett, Dent, Linstead, *J. Chem. Soc.*, 1936, 1719.

Linstead, Robertson, *ibid.*, 1736.

### Phthalonic Acid (o-Carboxybenzoylformic acid)



$\psi$ -Form

$\text{C}_9\text{H}_6\text{O}_5$  MW, 194

Cryst. +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd.  $146^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , pet. ether. Ox.  $\rightarrow$  phthalic acid.

$\alpha$ -Me ester:  $\text{C}_{10}\text{H}_8\text{O}_5$ . MW, 208. Cryst. +  $1\text{H}_2\text{O}$ . M.p.  $79-81^\circ$ .  $k = 1.5 \times 10^{-4}$  at  $25^\circ$ .

*Di-Me ester*:  $\text{C}_{11}\text{H}_{10}\text{O}_5$ . MW, 222. Cryst. from  $\text{MeOH}$ . M.p.  $66-8^\circ$ .

$\alpha$ -Amide:  $\alpha$ -phthalonamic acid.  $\text{C}_9\text{H}_7\text{O}_4\text{N}$ . MW, 193. Prisms +  $1\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd.  $178-9^\circ$  decomp.

$\beta$ -Amide:  $\beta$ -phthalonamic acid. M.p.  $155^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ .

*Anhydride*:  $\text{C}_9\text{H}_4\text{O}_4$ . MW, 176. Needles

from  $\text{Ac}_2\text{O}$ . M.p.  $185-6^\circ$  ( $190-1^\circ$ ) decomp. (rapid heat.). Spar. sol.  $\text{Et}_2\text{O}$ .

*Anilide*: phthalonanilic acid.  $\text{C}_{15}\text{H}_{11}\text{O}_4\text{N}$ . MW, 269. Needles from  $\text{CHCl}_3$ . M.p.  $176^\circ$ .

*Dianilide*: phthalonanilide.  $\text{C}_{21}\text{H}_{16}\text{O}_3\text{N}_2$ . MW, 344. Needles from  $\text{EtOH}$ . M.p.  $206-8^\circ$ .

*$\psi$ -Acetyl*: m.p.  $185-6^\circ \rightarrow$  phthalic anhydride + acetic acid.

*$\psi$ -Chloride*:  $\text{C}_9\text{H}_5\text{O}_4\text{Cl}$ . MW, 212.5. M.p.  $133^\circ$ .

*$\psi$ -Dichloride*:  $\text{C}_9\text{H}_4\text{O}_3\text{Cl}_2$ . MW, 231. M.p.  $70^\circ$ . B.p.  $120^\circ/12$  mm.

*$\psi$ -Phenylhydrazone*: m.p.  $171-2^\circ$ .

Tscherniac, *J. Chem. Soc.*, 1916, 109, 1236 (*Bibl.*).

Cornillot, *Ann. Chim.*, 1927, 7, 227; *Compt. rend.*, 1924, 179, 274.

Kuroda, Perkin, *J. Chem. Soc.*, 1923, 123, 2106.

Fuson, *J. Am. Chem. Soc.*, 1926, 48, 1093.

Gabriel, Colman, *Ber.*, 1900, 33, 999.

**Phthalonitrile** (*Phthalic acid dinitrile, o-dicyanobenzene*)



$\text{C}_8\text{H}_4\text{N}_2$

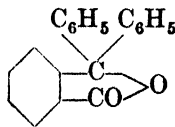
MW, 128

Needles from  $\text{H}_2\text{O}$ . M.p.  $141^\circ$  ( $140^\circ$ ). Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin. Volatile in steam. Slowly hyd. by hot  $\text{HCl}$  to the acid.

Braun, Tscherniac, *Ber.*, 1907, 40, 2710.

I.C.I., E.P., 413,639, (*Chem. Abstracts*, 1935, 29, 178).

**Phthalophenone** (*Diphenylphthalide, triphenylcarbinol-o-carboxylic lactone*)



$\text{C}_{20}\text{H}_{14}\text{O}_2$

MW, 286

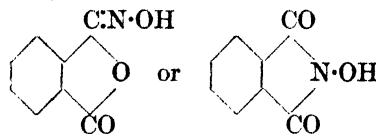
Leaflets from  $\text{EtOH}$ . M.p.  $115^\circ$ . B.p. above  $400^\circ$  decomp.  $\text{H}_2\text{SO}_4 \rightarrow$  greenish-yellow col.  $\rightarrow$  violet on heating.

Heller, *Ber.*, 1912, 45, 667.

Copisarow, *J. Chem. Soc.*, 1917, 111, 18.

Scheiber, *Ann.*, 1912, 389, 124.

**Phthaloxime** (*Isonitrosophthalide, N-hydroxyphthalimide*)



$\text{C}_8\text{H}_5\text{O}_3\text{N}$

MW, 163

(i) Needles from  $\text{EtOH}$ . M.p.  $220-6^\circ$  decomp. (rapid heat. in sealed tube). Sol. hot  $\text{EtOH}$ , hot  $\text{Me}_2\text{CO}$ . Mod. sol. hot  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*Acetyl deriv.*: m.p.  $183-5^\circ$ .

*Benzoyl deriv.*: m.p.  $171.5^\circ$ .

*O-Me*:  $\text{C}_9\text{H}_7\text{O}_3\text{N}$ . MW, 177. Needles from  $\text{EtOH}$ . M.p.  $133^\circ$ .

*O-Et*:  $\text{C}_{10}\text{H}_9\text{O}_3\text{N}$ . MW, 191. Prisms. M.p.  $95-100^\circ$ .

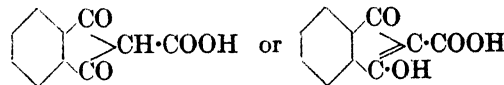
(ii) Yellow needles from  $\text{EtOH}$ . M.p.  $220-6^\circ$  decomp. (rapid heat. in sealed tube).

Acetyl and benzoyl derivs., *O-Me* and *O-Et* ethers identical with corresponding derivs. of (i).

Basler Chem. Fabrik, D.R.P., 130,681, (*Chem. Zentr.*, 1902, I, 1184).

Brady, Baker, Goldstein, Harris, *J. Chem. Soc.*, 1928, 538.

**Phthaloylacetic Acid** (*1:3-Diketohydrindene-2-carboxylic acid*)



$\text{C}_{10}\text{H}_6\text{O}_4$

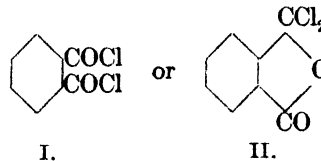
MW, 190

Unstable, readily losing  $\text{CO}_2 \rightarrow$  1:3-diketohydrindene.

*Et ester*:  $\text{C}_{12}\text{H}_{10}\text{O}_4$ . MW, 218. Yellow needles. M.p.  $75-8^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin. Insol.  $\text{H}_2\text{O}$ . Sol. alkalis  $\rightarrow$  enol salts.  $\text{FeCl}_3 \rightarrow$  red col. with  $\text{EtOH}$  sol.

Gabriel, Neumann, *Ber.*, 1893, 26, 953.

**Phthaloyl chloride** (*3:3-Dichlorophthalide*)



I.

II.

$\text{C}_8\text{H}_4\text{O}_2\text{Cl}_2$

MW, 203

I.

F.p.  $12^\circ$ . M.p.  $15-16^\circ$ . B.p.  $281.1^\circ$ ,  $275.4^\circ/726$  mm.,  $47^\circ/130$  mm.  $D_4^{20}$  1.4089.  $n_D^{20}$  1.571. Heat of comb.  $\text{C}_p$  802.03 Cal.,  $\text{C}_v$  802.05 Cal.



## II.

M.p. 88–9°. B.p. 275.2°/719.8 mm.

v. Braun, Kaiser, *Ber.*, 1922, **55**, 1305.

Ott, *Organic Syntheses*, 1931, **XI**, 88.

Garner, Sugden, *J. Chem. Soc.*, 1927, 2877.

Monsanto, E.P., 418,162, (*Chem. Abstracts*, 1935, **29**, 1436); U.S.P., 1,906,761, (*Chem. Abstracts*, 1933, **27**, 3484).

## Phthaloyl fluoride



$C_8H_4O_2F_2$

MW, 170

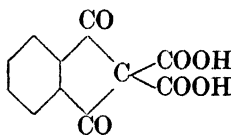
Plates from pet. ether. M.p. 42–3°. B.p. 224–6°/760 mm., 142°/65 mm., 135°/15 mm. Lachrymatory.  $H_2O \rightarrow$  phthalic anhydride.

Dunn, Davies, Hambly, Paul, Semmens, *J. Chem. Soc.*, 1933, 15.

## Phthaloylglycine.

See Phthaliminoacetic Acid.

**Phthaloylmalonic Acid** (1 : 3-Diketohydrindene-2 : 2-dicarboxylic acid)



$C_{11}H_6O_6$

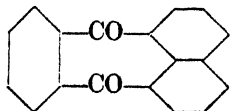
MW, 234

*Di-Et ester* :  $C_{15}H_{14}O_6$ . MW, 290. M.p. 74–5°. Sol.  $Et_2O$ , hot  $EtOH$ .  $D_4^{25}$  1.1896.  $n_D^{25}$  1.541.

*Et ester-nitrile* :  $C_{13}H_8O_4N$ . MW, 243. (i) M.p. 190–2°. Spar. sol.  $CHCl_3$ ,  $C_6H_6$ . (ii) M.p. 140–1°.

Scheiber, Hofer, *Ber.*, 1920, **53**, 898.

## 1 : 8-Phthaloylnaphthalene



$C_{18}H_{10}O_2$

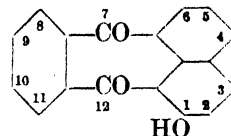
MW, 258

Needles from  $EtOH$ . M.p. 178°.  $H_2SO_4 \rightarrow$  yellow col. with green fluor.

*Mono-dinitrophenylhydrazone* : m.p. 265°.

Rieche, Sauthoff, Müller, *Ber.*, 1932, **65**, 1380.

## 1 : 8-Phthaloyl-2-naphthol (1-Hydroxy-7 : 12-dihydropleiadenedione-7 : 12)



$C_{18}H_{10}O_3$

MW, 274

Yellowish-green needles from chlorobenzene. M.p. 196°. Forms no phenylhydrazone.

*Acetyl deriv.* : m.p. 216°.

*Benzoyl deriv.* : m.p. 213°.

*7-Oxime* : m.p. 242°. *Acetyl deriv.* : m.p. 237°.

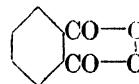
*12-Oxime* : m.p. 262°. *Acetyl deriv.* : m.p. 155–60°.

*Me ether* :  $C_{19}H_{12}O_3$ . MW, 288. M.p. 205°.

*Et ether* :  $C_{20}H_{14}O_3$ . MW, 302. Yellow leaflets from  $Et_2O$ . M.p. 163.5°.

Riche, Sauthoff, Müller, *Ber.*, 1932, **65**, 1379.

## Phthaloyl peroxide



$C_8H_4O_4$

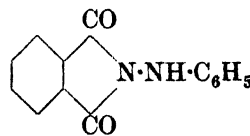
MW, 164

Cryst. M.p. 133.5° decomp. Explodes at 136°. Insol. ord. org. solvents.

Pechmann, Vanino, *Ber.*, 1894, **27**, 1511.

Baeyer, Villiger, *Ber.*, 1901, **34**, 762.

## Phthaloylphenylhydrazine (N-Anilino-phthalimide)



$C_{14}H_{10}O_2N_2$

MW, 238

(i) Yellow prisms from  $Me_2CO$ . M.p. 184°.  $D_4^{25}$  1.356.

(ii) Plates from  $Me_2CO$ . Does not melt.  $D_4^{27}$  1.354. Heat  $\rightarrow$  (i).

Sol.  $CHCl_3$ ,  $Me_2CO$ , hot  $EtOH$ , hot.  $AcOH$ . Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ , ligroin.

Scheiber, *Ann.*, 1912, **389**, 152.

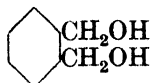
## Phthaloylpyridine.

See Anthrapyridinequinone.

## Phthaloylquinoline.

See Anthraquinolinequinone.

**Phthalyl Alcohol** (*o*-Xylylene glycol,  $\alpha$ : $\alpha$ -*o*-xylenediol, 1:2-di-(hydroxymethyl)-benzene,  $\omega\omega'$ -dihydroxy-*o*-xylene)

C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>

MW, 138

Plates from Et<sub>2</sub>O. M.p. 64.2–64.8° (62.5°). Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>.

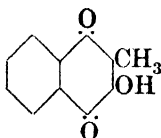
*Diacetyl*: m.p. 37°.

*Di-Et ether*: C<sub>12</sub>H<sub>18</sub>O<sub>2</sub>. MW, 194. Oil. B.p. 246–8°/738 mm.

Perkin, *J. Chem. Soc.*, 1888, 53, 7.

Wislicenus, Penndorf, *Ber.*, 1910, 43, 1837.

**Phthiocol** (3-Hydroxy-2-methyl-1:4-naphthoquinone)

C<sub>11</sub>H<sub>8</sub>O<sub>3</sub>

MW, 188

Pigment from tubercle bacilli. Yellow prisms from MeOH. Aq. M.p. 173–4°. Volatile in steam. Sol. dil. alkalis with intense red col. Ox. → phthalic acid.

*Acetyl deriv.*: m.p. 106–7°. Sol. alkalis to bright red sols.

*Monoxime*: m.p. 199–200°.

*Di-nitrophenylhydrazone*: does not melt below 270°.

*Me ether*: C<sub>12</sub>H<sub>10</sub>O<sub>3</sub>. MW, 202. M.p. 93°.

Madinaveitia, *Chem. Abstracts*, 1935, 29, 5438.

Newman, Crowder, Anderson, *J. Biol. Chem.*, 1934, 105, 279.

Ball, *J. Biol. Chem.*, 1934, 106, 515.

Anderson, Newman, *J. Biol. Chem.*, 1934, 103, 405.

### Phthioic Acid

C<sub>25</sub>H<sub>51</sub>·COOHC<sub>26</sub>H<sub>52</sub>O<sub>2</sub>

MW, 396

Obtained from human tubercle bacilli. M.p. 20–1°.  $D_4^{25}$  0.8763.  $n_D^{25}$  1.4628.  $[\alpha]_D + 12.56^\circ$ . The hydrocarbon chain is branched.

*Me ester*: C<sub>27</sub>H<sub>54</sub>O<sub>2</sub>. MW, 410. B.p. 175–8°/0.05 mm., 158°/0.03 mm.  $D_4^{25}$  0.8620.  $n_D^{25}$  1.4550.  $[\alpha]_D^{25} + 12.2^\circ$ .

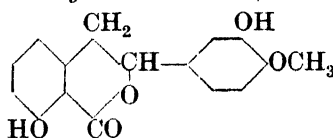
*Amide*: C<sub>26</sub>H<sub>53</sub>ON. MW, 395. Plates from EtOH. M.p. 45°.

Dict. of Org. Comp.—III.

*Methylamide*: C<sub>27</sub>H<sub>55</sub>ON. MW, 409. M.p. 27°.

Spielman, Anderson, *J. Biol. Chem.*, 1936, 112, 759 (*Bibl.*).

**Phyllo dulcin** (8:3'-Dihydroxy-4'-methoxy-3-phenyl-3:4-dihydroisocoumarin)

C<sub>16</sub>H<sub>14</sub>O<sub>5</sub>

MW, 286

d-.

Occurs in leaves of *Hydrangea thumbergii*, Sieb. M.p. 120°.  $[\alpha]_D + 67.7$  to 69.7°. It was originally thought that a natural preparation consisted of a mixture of *d*-phyllo dulcin + another compound which was given the name *d*-isophyllo dulcin. The mixture has now been shown to consist of *d* + *dl*-phyllo dulcin and accordingly isophyllo dulcin does not exist.

*Diacetyl deriv.*: m.p. 148–9°.

*Dibenzoyl deriv.*: m.p. 183–183.5°.

dl-.

M.p. 131°.

*Di-Me ether*: C<sub>18</sub>H<sub>18</sub>O<sub>5</sub>. MW, 314. (i) Needles from EtOH. M.p. 105°. Labile. (ii) M.p. 125°. Stable.

Maniwa, *Chem. Abstracts*, 1924, 18, 2694.  
Asahina, Asano, *Ber.*, 1931, 64, 1253.

**Phyllo merol** (6:7-Dihydroxy-2-methylnaphthalene)

C<sub>11</sub>H<sub>10</sub>O<sub>2</sub>

MW, 174

Cryst. M.p. 161–2°. FeCl<sub>3</sub> → green col. in MeOH.

*Me ether*: C<sub>12</sub>H<sub>12</sub>O<sub>2</sub>. MW, 188. M.p. 119–22°.

*Di-Me ether*: C<sub>13</sub>H<sub>14</sub>O<sub>2</sub>. MW, 202. Needles. M.p. 98–100°. *Picrate*: m.p. 120–1°.

*Methylene ether*: see Podophyllomerol.

Borsche, Niemann, *Ber.*, 1932, 65, 1633;  
*Ann.*, 1933, 502, 268.

**Phyllo meronic Acid** (6:7-Dihydroxy-2-methylnaphthalene-3-carboxylic acid, phyllo merol-3-carboxylic acid, 6:7-dihydroxy-3-methyl-2-naphthoic acid)

C<sub>12</sub>H<sub>10</sub>O<sub>4</sub>MW, 218  
31

Leaflets from MeOH. M.p. 243–4°.  $\text{FeCl}_3 \rightarrow$  deep blue col.

6 : 7-Diacetyl : m.p. 220°.

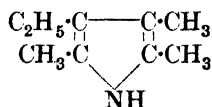
Me ester :  $\text{C}_{13}\text{H}_{12}\text{O}_4$ . MW, 232. Needles from MeOH. M.p. 186–7°.  $\text{FeCl}_3 \rightarrow$  green col.

Di-Me ether :  $\text{C}_{14}\text{H}_{14}\text{O}_4$ . MW, 246. Needles from MeOH. M.p. 223–5°. Me ester :  $\text{C}_{15}\text{H}_{16}\text{O}_4$ . MW, 260. Needles from MeOH. M.p. 125–6°.

Methylene ether : *see* Podophyllomeric Acid.

Borsche, Niemann, *Ber.*, 1932, 65, 1633; *Ann.*, 1932, 499, 68.

**Phyllopyrrole** (2 : 3 : 5-Trimethyl-4-ethylpyrrole)



$\text{C}_9\text{H}_{15}\text{N}$  MW, 137

Leaflets from  $\text{Et}_2\text{O}$ . M.p. 66–7°. B.p. 213°/725 mm., 110–12°/35 mm., 92–3°/12 mm. Turns oily then resinous in air.

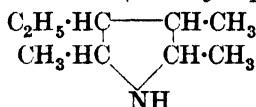
Picrate : m.p. 101–3°.

Siedel, *Z. physiol. Chem.*, 1935, 231, 167.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, 58, 712.

Colacicchi, *Atti accad. Lincei*, 1913, 21, i, 489.

**Phyllopyrrolidine** (Tetrahydrophyllopyrrole)



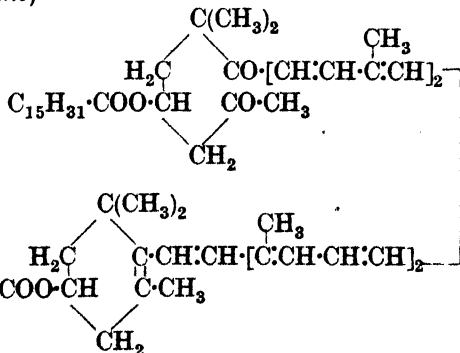
$\text{C}_9\text{H}_{19}\text{N}$  MW, 141

B.p. 160–4°.  $D_4^{20}$  0.824.

1-Naphthylurea : m.p. 145°.

Willstätter, Asahina, *Ann.*, 1911, 385, 215.

**Physalien** (Zeaxanthin dipalmitate, physalin, physaliene)



$\text{C}_{72}\text{H}_{116}\text{O}_4$  MW, 1044

Occurs in berries of *Physalis* species. Red cryst. from  $\text{C}_6\text{H}_6$ -MeOH. M.p. 98.5–99.5°.

Winterstein, Ehrenberg, *Z. physiol. Chem.*, 1932, 207, 25.

Kuhn, Grundmann, *Ber.*, 1934, 67, 596.

Karrer, Solmssen, Walker, *Helv. Chim. Acta*, 1934, 17, 417.

Cholnoky, *Z. physiol. Chem.*, 1930, 189, 159.

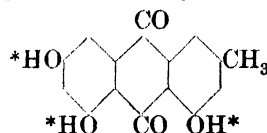
Kylin, *Z. physiol. Chem.*, 1927, 183, 229.

Kuhn, Wiegand, *Helv. Chim. Acta*, 1929, 12, 499.

**Physciaic Acid.**

*See* Physcione.

**Physcione** (Physciaic acid, parietin, chrysophyscin, rheumemodin methyl ether, frangulaemodin methyl ether, rheochrysidine)



$\text{C}_{16}\text{H}_{12}\text{O}_5$  MW, 284

Occurs in chrysarobin, rhubarb root and lichens. M.p. 207°. Sol.  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , Py, toluene. Spar. sol. AcOH, AcOEt. Insol. EtOH, MeOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ .

Tutin, Clewer, *J. Chem. Soc.*, 1912, 101, 294.

Eder, Hauser, *Helv. Chim. Acta*, 1925, 8, 140 (*Bibl.*).

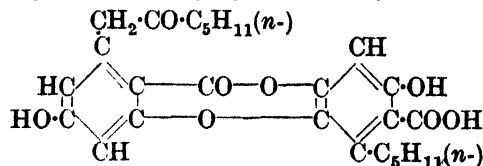
Hesse, *Ann.*, 1917, 413, 368.

\* One of these groups is  $\text{OCH}_3$  but which one is not known.

**Physodalic Acid.**

*See* Physodic Acid.

**Physodic Acid** (Physodalic acid)



$\text{C}_{26}\text{H}_{40}\text{O}_8$  MW, 470

Occurs in *Parmelia physodes*, and *Cetraria islandica*. Needles from MeOH.Aq. M.p. 205°.  $\text{FeCl}_3 \rightarrow$  violet col.  $\text{Zn} + \text{NaOH} \rightarrow$  atranol + orcinol.

Diacetyl deriv. : m.p. 153–153.5°.

Oxime : m.p. 209–10°.

Phenylhydrazone : m.p. 265° decomp.

Me ester :  $\text{C}_{27}\text{H}_{42}\text{O}_8$ . MW, 484. Prisms from EtOH. M.p. 156–7°. Me ether :  $\text{C}_{28}\text{H}_{44}\text{O}_8$ .

MW, 498. Leaflets from AcOH. M.p. 117–19°. Diacetyl deriv.: m.p. 114–15°.

Anilide: m.p. 260° decomp.

Asahina, Nogami, *Ber.*, 1935, **68**, 1500; 1934, **67**, 805.

Koller, Locker, *Monatsh.*, 1931, **58**, 209.

*Note.*—Some doubt exists as to the identity of physodic with physodylic acid, *q.v.* Hesse, *J. prakt. Chem.*, 1915, **92**, 439, identifies it with capraric acid.

### Physodylic Acid (Isidic Acid)

$C_{23}H_{24}O_6$  MW, 396

Occurs in *Evernia furfuracea*, Linn. Needles from AcOH. M.p. 196° (169–70°). Spar. sol. Et<sub>2</sub>O.

Hesse, *J. prakt. Chem.*, 1915, **92**, 437.

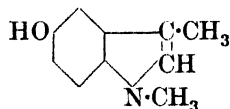
### Physostigmine.

See Eserine.

### Physostigmine oxide.

See Geneserine.

**Physostigmol** (5-Hydroxy-1 : 3-dimethyl-indole)



$C_{10}H_{11}ON$  MW, 161

Needles by sublimation, m.p. 114° (100°). Plates by cryst., m.p. indefinite.

*Me ether*:  $C_{11}H_{13}ON$ . MW, 175. Leaflets from MeOH. M.p. 60–1°.

*Et ether*:  $C_{12}H_{15}ON$ . MW, 189. M.p. 86° (85°). *Picrate*: m.p. 95°.

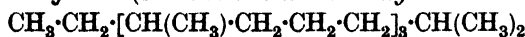
Stedman, *J. Chem. Soc.*, 1924, **125**, 1373.

Julian, Piki, *J. Am. Chem. Soc.*, 1935, **57**, 566.

Keimatsu, Sugasawa, *Chem. Abstracts*, 1928, **22**, 3163.

Straus, *Ann.*, 1914, **406**, 337.

### Phytane (3 : 7 : 11 : 15-Tetramethylhexadecane)

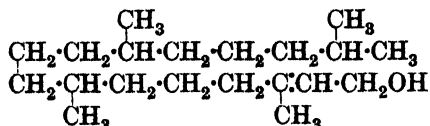


$C_{20}H_{42}$  MW, 282

B.p. 179.5°/15 mm., 169.5°/9.5 mm. Sol. pet. ether, hot EtOH, hot AcOH.  $D_4^{20}$  0.803.

Karrer, *Helv. Chim. Acta*, 1929, **12**, 906.

**Phytol** (3 : 7 : 11 : 15-Tetramethyl-2-hexadecanol)



$C_{20}H_{40}O$  MW, 296

Decomp. product of chlorophyll. B.p. 202.5–204°/10 mm. Sol. ord. org. solvents.  $D_4^{20}$  0.8497.  $n_D^{20}$  1.4595.

*Urethane*: m.p. 25.8–28.9°.

*Acid phthalate*: *Ag salt*, m.p. 117–19°.

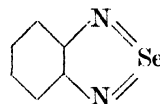
*Pyruvate*: semicarbazone, needles from MeOH. M.p. 72–5°.

Fischer, Löwenberg, *Ann.*, 1929, **475**, 183.

### Phytosterol.

Generic name for the plant sterols. See e.g., Fucosterol, Sitosterol, and Stigmasterol.

### Piaselenole



$C_6H_4N_2Se$  MW, 183

Needles with odour of quinoline. M.p. 76°. B.p. 246°. Sol. ord. org. solvents. Spar. sol. H<sub>2</sub>O.

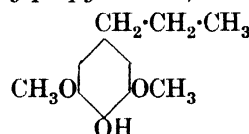
*B.HClO<sub>4</sub>*: canary-yellow cryst. from conc. HClO<sub>4</sub>. Hyd. by H<sub>2</sub>O.

Battegay, Véchet, *Bull. soc. chim.*, 1925, **37**, 1281.

Heinemann, E.P., 3,042; D.R.P., 261,412, (*Chem. Abstracts*, 1913, **7**, 3200).

Hinsberg, *Ber.*, 1889, **22**, 2897; *J. prakt. Chem.*, 1916, **94**, 182.

**Picamar** (4-Propyl-2 : 6-dimethoxyphenol, 5-propylpyrogallol-1 : 3-dimethyl ether, 4-hydroxy-3 : 5-dimethoxy-propylbenzene)



$C_{11}H_{16}O_3$  MW, 196

Oil. B.p. 285°.

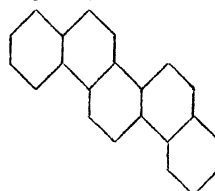
*Acetyl*: cryst. from EtOH. M.p. 87°. Br in CCl<sub>4</sub> → dibromo deriv., m.p. 101–2°.

Mauthner, *J. prakt. Chem.*, 1921, **102**, 37.

### Picein.

See Piceoside.

**Picene** (1 : 2 : 7 : 8-Dibenzphenanthrene, β : β-dinaphthylene-ethylene)



$C_{22}H_{14}$  MW, 278

Colourless cryst. from Py or boiling xylene. M.p. 365–6°. B.p. 518–20°. Sublimes. Spar. sol. boiling  $C_6H_6$ ,  $CHCl_3$ , AcOH. Fluoresces pale blue in ultra-violet light. Sol. conc.  $H_2SO_4$   $\rightarrow$  intense green col.

Dibromide: m.p. 295°.

2:7-Dinitroanthraquinone add. comp.: m.p. 299–300°.

Ruzicka *et al.*, *Helv. Chim. Acta*, 1934, 17, 200, 470.

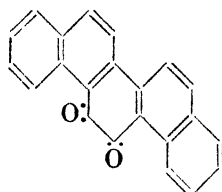
Winterstein, Schön, Vetter, *Z. physiol. Chem.*, 1934, 230, 158.

Friedmann, *Ber.*, 1916, 49, 277.

Meyer, Hofmann, *Monatsh.*, 1916, 37, 715.

Bamberger, Chattaway, *Ann.*, 1895, 284, 60.

### Picenequinone



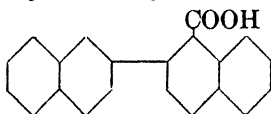
$C_{22}H_{12}O_2$

MW, 308

Red needles or leaflets from boiling AcOH. Sublimes on slow heating. Decomp. on rapid heating. Dist. with Zn dust  $\rightarrow$  picene. Dist. over lead foil in vacuo  $\rightarrow$  picylene ketone.

Meyer, Hofmann, *Monatsh.*, 1916, 37, 681, 721.

**Picenic Acid** (2- $\beta$ -Naphthyl-1-naphthoic acid, 2:2'-dinaphthyl-1-carboxylic acid)



$C_{21}H_{14}O_2$

MW, 298

Cryst. from EtOH. M.p. 201°. Sol. EtOH,  $C_6H_6$ ,  $CHCl_3$ . Conc.  $H_2SO_4$   $\rightarrow$  picylene ketone. Dist. with CaO  $\rightarrow$  2:2'-dinaphthyl.

Bamberger, Chattaway, *Ann.*, 1895, 284, 71.

**Piceoside** (*Amelarioside*, *p*-hydroxyacetophenone- $\beta$ -glucoside, *picein*, *salinigrin*)



$C_{14}H_{18}O_7$

MW, 298

Constituent of various willow barks, *Amelanchier vulgaris*, Moench., *Picea excelsa*, Link., *Salix discolor*, Muhl. Cryst. +  $1H_2O$  from  $H_2O$ .

M.p. 195°.  $[\alpha]_D -86.5^\circ$ . Sol. EtOH,  $Et_2O$ , AcOH. Spar. sol.  $H_2O$ . Dil. acids or emulsin  $\rightarrow$  *p*-hydroxyacetophenone + glucose.

Tetra-acetyl: m.p. 172–3°.

Oxime: m.p. 228°.

Phenylhydrazone: m.p. 185°.

Semicarbazone: m.p. 220°.

Ramart-Lucas, Rabaté, *Compt. rend.*, 1933, 196, 1493.

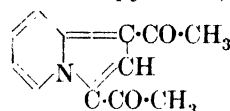
Jowett, *J. Chem. Soc.*, 1932, 721.

Rabaté, *Bull. soc. chim. biol.*, 1930, 12, 146, 332, 441, 965.

Bargellini, Leone, *Atti accad. Lincei*, 1925, 2, 35.

Mauthner, *J. prakt. Chem.*, 1913, 88, 764.

**Picolide** (1:3-Diacetoindolizine, 1:3-diacetopyrrocolin, 1:3-diacetopyrindole)



$C_{12}H_{11}O_2N$

MW, 201

Needles from  $H_2O$ . M.p. 176°. B.p. 360° (slight decomp.). Sol.  $CHCl_3$ , AcOH, Py. Mod. sol. cold EtOH. Insol.  $Et_2O$ . Sol. in conc. HCl re-ppd. by  $H_2O$ .  $CHCl_3$  sol. adds Br. Decolourises  $KMnO_4$ . Aq. Heat with 25% HCl  $\rightarrow$  pyrrocolin.

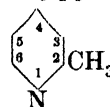
Monoxime: needles from EtOH or  $H_2O$ . M.p. 244°.

Monophenylhydrazone: yellow needles from EtOH. Aq. M.p. 168°.

Monosemicarbazone: yellow plates from EtOH. M.p. 233°.

Tschitschibabin, Stepanow, *Ber.*, 1929, 62, 1068.

$\alpha$ -Picoline (2-Methylpyridine)



$C_6H_7N$

MW, 93

Oil with strong unpleasant odour. B.p. 129°.  $D_4^{15} 0.9497$ .  $n_D^{15} 1.5029$ . Misc. with most solvents. Heat of comb.  $C_6H_7N$  815.4 Cal.  $k = 3.2 \times 10^{-8}$  at 25°. Oxidised by boiling  $KMnO_4$ . Aq. or  $SeO_2$   $\rightarrow$  picolinic acid. Reduced by Na in EtOH or by H (+ Pd or Pt)  $\rightarrow$   $\alpha$ -pipecoline. Forms numerous complex salts.

$B, HCl$ : hygroscopic cryst. +  $\frac{1}{2}H_2O$ . M.p. anhyd. 200°.

$B, HBr, Br$ : red cryst. M.p. 76°.

$B, HBr, HgBr_2$ : needles. M.p. 88°.

$B, HI, I_2$ : dark brown cryst. M.p. 44°.  
 $B, H, AuCl_4$ : cryst. M.p. 183-4° (167-8°).  
 $B_2, 3HgCl_2$ : needles. M.p. 170-2°.  
 $B, HCl, 2HgCl_2$ : prisms from  $H_2O$ . M.p. 154°.  
 $B_2, H_2PtCl_6$ : cryst. M.p. 216-17° (195°) decomp.  
 Methiodide: needles from EtOH. M.p. 226-8°.  
 Chloroplatinate: m.p. 225-6°.  
 Ethobromide: m.p. 97°.  
 Ethiodide: m.p. 123°.  
 Picrate: needles. M.p. 169-71° (163°).  
 Styphnate: m.p. 180°.

(Cartwright, Errera, *Compt. rend.*, 1935, 200, 914.

Henze, *Ber.*, 1934, 67, 750.

Tartarini, Samaja, *Ann. chim. applicata*, 1933, 23, 351.

Borisov, *Ber.*, 1930, 63, 2278.

I.G., E.P., 283,163, (*Chem. Abstracts*, 1928, 22, 3892).

Wilkie, Shaw, *J. Soc. Chem. Ind.*, 1927, 46, 469r.

Heap, Jones, Speakman, *J. Am. Chem. Soc.*, 1921, 43, 1936.

**$\beta$ -Picoline (3-Methylpyridine).**

Oil with sweetish odour. B.p. 143.8°.  $D_4^{25}$  0.9515.  $n_D^{25}$  1.5043. Misc. with most solvents. Heat of comb.  $C_v$  812.4 Cal.  $k = 1.1 \times 10^{-8}$  at 25°. Forms numerous complex salts.  $KMnO_4 \rightarrow$  nicotinic acid. Red.  $\rightarrow$   $\beta$ -pipercoline.  $NaNH_2$  in boiling xylene  $\rightarrow$  1-amino- $\beta$ -picoline, m.p. 24°. Br in  $CCl_4 \rightarrow$  complex salt of bromopicoline, m.p. 200°.

$B, HCl, 2HgCl_2$ : cryst. from EtOH. M.p. 147-9°.

$B_2, ZnCl_2$ : needles. M.p. 158°.

$B, H, AuCl_4$ : needles. M.p. 182-4°.

$B_2, H_2PtCl_6$ : prisms +  $H_2O$ . M.p. anhyd. 202°.

Methiodide + 2I: light brown leaflets from EtOH. M.p. 36°.

Picrate: m.p. 149-50°.

Styphnate: m.p. 153-4°.

Aldred, Lyons, *Chem. Abstracts*, 1931, 25, 3345.

See also second and last references above.

**$\gamma$ -Picoline (4-Methylpyridine).**

B.p. 143.1°.  $D_4^{15}$  0.9571.  $n_D^{15}$  1.5064. Heat of comb.  $C_v$  816 Cal.  $k = 1.1 \times 10^{-8}$  at 25°.  $KMnO_4 \rightarrow$  isonicotinic acid. Red.  $\rightarrow$   $\gamma$ -pipercoline.

$B, 2HgCl_2$ : needles. M.p. 128-9°.

$B, H, AuCl_4$ : prisms from  $H_2O$ . M.p. 205° decomp.

$B_2, H_2PtCl_6$ : leaflets. M.p. 231-44° according to rate of heating.

Methiodide + 2I: light brown plates from EtOH. M.p. 101°.

Methiodide + 4I: steel-blue needles. M.p. 63°.

Methiodide + 6I: dark green needles. M.p. 81.5°.

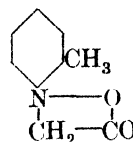
Picrate: m.p. 167°.

Bailey, McElvain, *J. Am. Chem. Soc.*, 1930, 52, 1633.

I.G., E.P., 283,163, (*Chem. Abstracts*, 1928, 22, 3892).

Meisenheimer, *Ann.*, 1920, 420, 197.

**$\alpha$ -Picoline-betaïne**



$C_8H_9O_2N$

MW, 151

Needles from EtOH-Et<sub>2</sub>O. Decomp. at 162° (turns brown above 100°). Very hygroscopic. Sol. EtOH.  $H_2O$  sol. reacts neutral.

$B, HCl$ : m.p. 188° decomp.

$B_2, H_2PtCl_6$ : prisms + 2 $H_2O$  from HCl.Aq. M.p. 212° decomp.

Kirpal, *Monatsh.*, 1910, 31, 976.

**$\beta$ -Picoline-betaïne.**

Leaflets + 1 $H_2O$  from EtOH-Et<sub>2</sub>O. Sol.  $H_2O$ , EtOH.

$B, HCl$ : prisms. M.p. 189° decomp.

$B_2, H_2PtCl_6$ : plates from  $H_2O$ . Decomp. at 222°.

Krüger, *J. prakt. Chem.*, 1891, 43, 364, 370.

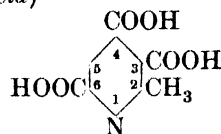
**Picoline-carboxylic Acid.**

See Homonicotinic Acid, Methylnicotinic Acid, and Methylpicolinic Acid.

**Picoline-dicarboxylic Acid.**

See 2-Methylcinchomeronic Acid and Uvitonic Acid.

**$\alpha$ -Picoline-3 : 4 : 6-tricarboxylic Acid (6-Methylberberonic acid, 2-methylpyridine-3 : 4 : 6-tricarboxylic acid)**



$C_9H_7O_6N$

MW, 225

Prisms or plates + 3 $H_2O$  from  $H_2O$ . M.p. anhyd. 226° decomp. (sealed tube). Very sol.

hot  $\text{H}_2\text{O}$ . Sol. EtOH, AcOH. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin. On standing several days with AcOH  $\rightarrow$   $\alpha$ -picoline-3 : 4-dicarboxylic acid. Conc. aq. sol. + ferrous ammonium sulphate  $\rightarrow$  dark red col. + ppt. Warmed with Cu acetate  $\rightarrow$  bluish-green ppt.

Anilide-phenylimide :  $\text{C}_{21}\text{H}_{15}\text{O}_3\text{N}_3$ . MW, 357. Needles. M.p.  $237^\circ$ .

Lawson, Perkin, Robinson, *J. Chem. Soc.*, 1924, 125, 631, 638.

Cf. Mumm, Hüneke, *Ber.*, 1918, 51, 155.

**$\alpha$ -Picoline-3 : 5 : 6-tricarboxylic Acid** (2-Methylpyridine-3 : 5 : 6-tricarboxylic acid, 6-methylcarbodinicotinic acid).

Cryst. +  $1\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . Turns yellow at  $170^\circ$ . M.p.  $226^\circ$  decomp. The acid or its K salt on heating to  $150^\circ \rightarrow \alpha$ -picoline-3 : 5-dicarboxylic acid.  $\text{FeCl}_3$ . Aq.  $\rightarrow$  yellow ppt.

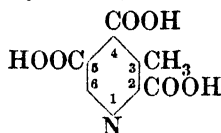
Weber, *Ann.*, 1887, 241, 6.

**$\alpha$ -Picoline-4 : 5 : 6-tricarboxylic Acid** (2-Methylpyridine-4 : 5 : 6-tricarboxylic acid, 6-methylcarbocinchomeric acid).

Prisms or plates from  $\text{H}_2\text{O}$ . Decomp. slowly on heating (turns yellow at  $210^\circ$ , dark brown to black at  $280^\circ$ ). Sol.  $\text{H}_2\text{O}$ . Very spar. sol. EtOH,  $\text{Et}_2\text{O}$ . Stable to boiling AcOH.  $\text{FeSO}_4$ . Aq.  $\rightarrow$  red col.

Mumm, Hüneke, *Ber.*, 1918, 51, 157.

**$\beta$ -Picoline-2 : 4 : 5-tricarboxylic Acid** (3-Methylberberonic acid, 3-methylpyridine-2 : 4 : 5-tricarboxylic acid)



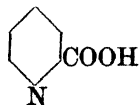
$\text{C}_9\text{H}_7\text{O}_6\text{N}$  MW, 225

Prisms +  $1\text{H}_2\text{O}$ . M.p.  $208^\circ$ . Sol. EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .  $\text{FeSO}_4$ . Aq.  $\rightarrow$  weak reddish-brown col.

Dobbie, Lauder, *J. Chem. Soc.*, 1902, 81, 151.

Dobbie, Marsden, *J. Chem. Soc.*, 1897, 71, 663.

**Picolinic Acid** (Pyridine-2-carboxylic acid)



$\text{C}_6\text{H}_5\text{O}_2\text{N}$  MW, 123

Needles from  $\text{H}_2\text{O}$ , EtOH or  $\text{C}_6\text{H}_6$ . M.p.  $136-7^\circ$ . Sublimes. Very sol. AcOH. Sol.

$\text{H}_2\text{O}$ . Mod. sol. EtOH. Spar. sol.  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ . Na in boiling EtOH  $\rightarrow$  hexahydronicolinic acid.  $\text{NaHg}$  in  $\text{H}_2\text{O} \rightarrow$  1-hydroxyadipic acid. Conc. alc. KOH at  $240^\circ \rightarrow$  pyridine. Zn dust + AcOH  $\rightarrow \alpha$ -picoline.

Me ester :  $\text{C}_7\text{H}_7\text{O}_2\text{N}$ . MW, 137. Hygroscopic cryst. M.p.  $14^\circ$ . B.p.  $232^\circ$ .

Et ester :  $\text{C}_8\text{H}_9\text{O}_2\text{N}$ . MW, 151. B.p.  $243^\circ$ ,  $122/13$  mm.

Phenyl ester :  $\text{C}_{12}\text{H}_9\text{O}_2\text{N}$ . MW, 199. M.p.  $82^\circ$ .

Anhydride : m.p.  $124^\circ$  (sealed tube).

Chloride :  $\text{C}_6\text{H}_5\text{ONCl}$ . MW, 141.5. M.p.  $46^\circ$ . B.p.  $160^\circ$  (slight decomp.)/10 mm.

Amide :  $\text{C}_6\text{H}_6\text{ON}_2$ . MW, 122. M.p.  $106.5^\circ$ .

Anilide : m.p.  $76^\circ$ .

p-Toluidide : m.p.  $104^\circ$ .

Nitrile : see 2-Cyanopyridine.

N-Me-betaïne : see Homarine.

Aurichloride : m.p.  $200^\circ$  ( $204^\circ$  decomp.).

Platinichloride : cryst. +  $2\text{H}_2\text{O}$ . M.p.  $215-16^\circ$ .

Brode, Bremer, *J. Am. Chem. Soc.*, 1934, 56, 993.

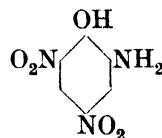
Hoppe-Seyler, *Z. physiol. Chem.*, 1933, 222, 105.

Meyer, *Rec. trav. chim.*, 1925, 44, 323.

Hess, Leibbrandt, *Ber.*, 1917, 50, 385.

Ley, Ficken, *ibid.*, 1132.

**Picramic Acid** (4 : 6-Dinitro-2-aminophenol)



$\text{C}_6\text{H}_5\text{O}_5\text{N}_3$  MW, 199

Dark red needles from EtOH, prisms from  $\text{CHCl}_3$ . M.p.  $169^\circ$ . Sol.  $\text{C}_6\text{H}_6$ , AcOH. Mod. sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Sol. to 0.14% in  $\text{H}_2\text{O}$  at  $22^\circ$ . Sol. alkalis  $\rightarrow$  reddish-brown col. Colourless sol. in conc. HCl.  $k = 2.5 \times 10^{-5}$  at  $0^\circ$ ,  $1.1 \times 10^{-4}$  at  $65^\circ$ . Forms salts with acids and bases. More toxic than picric acid. Intermediate for azo dyestuffs. Gives colour reactions with proteins, amino-acids and amines, but not with their salts.

N-Acetyl : needles from  $\text{H}_2\text{O}$ . M.p.  $201^\circ$ .

N-Benzoyl : greenish-yellow needles from AcOH or xylene. M.p.  $299-30^\circ$ .

N-p-Toluenesulphonyl : m.p.  $191^\circ$ . Py salt : m.p.  $203^\circ$ .

O-Acetyl : m.p.  $193^\circ$ .

O-Benzoyl : leaflets. M.p.  $218-19^\circ$ .

N-Benzyl : ochre prisms. M.p.  $139-40^\circ$ .

N-Me : orange scales from dil. EtOH. M.p. 144-5°.

N-di-Me : yellow micro-cryst. from AcOH. M.p. 218-20°.

O-Me ether : 4 : 6-dinitro-2-aminoanisole.  $C_7H_7O_5N_3$ . MW, 213. Violet needles from EtOH. Insol.  $H_2O$ .

Clayton, *J. Soc. Dyers Colourists*, 1930, **46**, 365, (*Chem. Abstracts*, 1931, **25**, 926).

Seyewetz, Blanc, *Chimie et Industrie*, 1930, **25**, 605.

Dehn, U.S.P., 1,472,791, (*Chem. Abstracts*, 1924, **18**, 400).

\* Pomeranz, *Chem.-Ztg.*, 1921, **45**, 865, (*Chem. Abstracts*, 1922, **16**, 163).

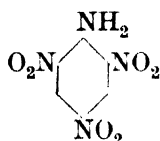
Egerer, *J. Biol. Chem.*, 1918, **35**, 565.

Aloy, Frébault, *Bull. soc. chim.*, 1904, **33**, 496.

Hofer, Jakob, *Ber.*, 1908, **41**, 3198.

Kym, *Ber.*, 1899, **32**, 1429.

#### Picramide (2 : 4 : 6-Trinitroaniline)



$C_6H_4O_6N_4$  MW, 228

Prisms from AcOH. M.p. 188° (192-5°). Sol.  $Me_2CO$ , AcOEt,  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ . Hot KOH  $\rightarrow$   $NH_3$  + picric acid.

N-Acetyl : 2 : 4 : 6-trinitroacetanilide. Needles from AcOH.Aq. M.p. about 230°.

N-Benzoyl : 2 : 4 : 6-trinitrobenzanilide. Needles from EtOH. M.p. 195-6°.

N-Me :  $C_7H_6O_6N_4$ . MW, 242. Yellow needles from EtOH. M.p. 114-8°.

N-Di-Me :  $C_8H_8O_6N_4$ . MW, 256. Yellow plates from  $C_6H_6$ . M.p. 138°.

N-Et :  $C_8H_8O_6N_4$ . MW, 256. Cryst. from  $C_6H_6$ . M.p. 84°.

N-Di-Et :  $C_{10}H_{12}O_6N_4$ . MW, 284. Prisms from  $C_6H_6$ . M.p. 163°.

N-Propyl :  $C_9H_{10}O_6N_4$ . MW, 270. Yellow needles. M.p. 59°.

N-Dipropyl :  $C_{12}H_{16}O_6N_4$ . MW, 312. Orange-red cryst. from  $Me_2CO-CHCl_3$ . M.p. 38°.

N-Isopropyl : yellow needles from EtOH-AcOH. M.p. 106-7°.

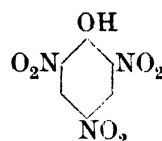
N-Phenyl : see 2 : 4 : 6-Trinitrodiphenylamine.

Hollemann, *Rec. trav. chim.*, 1930, **49**, 112.

James, Jones, Lewis, *J. Chem. Soc.*, 1920, **117**, 1273.

Weiss, Abeles, *Monatsh.*, 1932, **59**, 238.

#### Picric Acid (2 : 4 : 6-Trinitrophenol)



$C_6H_3O_7N_3$  MW, 229

Yellow leaflets from  $H_2O$ , prisms from  $Et_2O$ , plates from EtOH. Colourless cryst. from hot ligroin or conc. HCl. M.p. 122.5°. Sublimes with slow heat, deflagrates with rapid heat. Explosive, but salts are more so. Very bitter taste. Toxic.

Percentage solubilities :— $H_2O$ , 0.98 at 9°; 2.33 at 50°; 7.6 at 100°; MeOH, 16 at 16°, 40.2 at 50°; EtOH, 6.8 at 16°, 19.7 at 50°; AcOEt, 39.4 at 16°, 68.5 at 50°;  $Me_2CO$ , 123.3 at 16°, 220.5 at 50°;  $Et_2O$ , 2.64 at 16°, 4.0 at 50°;  $CHCl_3$ , 2.0 at 16°, 5.7 at 50°;  $C_6H_6$ , 7.5 at 16°, 29.5 at 50°; toluene, 12.2 at 16°, 27.8 at 50°;  $CCl_4$ , 0.06 at 16°, 0.35 at 50°;  $CS_2$ , 0.11 at 16°, 0.18 at 34°; Py, 27.62 at 16°, 58.9 at 50°. Forms add. comp. with 2Py, m.p. 144-5°. Stable to hot conc.  $H_2SO_4$  or HCl. Red.  $\rightarrow$  picramic acid  $\rightarrow$  2 : 6-diamino-4-nitrophenol  $\rightarrow$  2 : 4 : 6-triaminophenol. Cl or *aqua regia* or  $KClO_3 + HCl \rightarrow$  chloranil + chloropicrin.  $PCl_5 \rightarrow$  picryl chloride. Forms cryst. add. comps. with many bases, phenols, hydrocarbons, etc.

Molnar, *Compt. rend.*, 1935, **201**, 59.

Haid, Koenen, *Chem. Abstracts*, 1934, **28**, 2907.

I.C.I., E.P., 370,436, (*Chem. Abstracts*, 1933, **27**, 2965).

Muraour, *Bull. soc. chim.*, 1932, **51**, 1152.

Desvergnès, *Chimie et Industrie*, 1929, **22**, 451.

Benedict, *J. Biol. Chem.*, 1929, **82**, 1.

Olsen, Goldstein, *Ind. Eng. Chem.*, 1924, **16**, 66.

Robertson, Garner, *Proc. Roy. Soc.*, 1923, **103A**, 539.

Holliday, Badier, U.S.P., 1,413,914, (*Chem. Abstracts*, 1922, **16**, 2150).

Marqueyrol, Lorient, *Bull. soc. chim.*, 1919, **25**, 376.

Me ether : 2 : 4 : 6-trinitroanisole.  $C_7H_5O_7N_3$ . MW, 243. Colourless plates or leaflets from EtOH. M.p. 68°.

Et ether : 2 : 4 : 6-trinitrophenetole.  $C_8H_7O_7N_3$ . MW, 257. Colourless needles from EtOH. M.p. 78.5°. Sol.  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol. hot  $H_2O$ .



*Phenyl ether*: see 2:4:6-Trinitrodiphenyl Ether.

*Benzyl ether*:  $C_{18}H_{19}O_7N_3$ . MW, 319. Colourless prisms from  $C_6H_6$ . M.p. 147°.

*Acetyl*: colourless cryst. from  $Et_2O$ . M.p. 75-6°.

Tommasi, David, *Ann.*, 1873, **169**, 167;

*Compt. rend.*, 1873, **77**, 207.

Kumpf, *Ann.*, 1884, **224**, 131.

Jackson, Gazzolo, *Am. Chem. J.*, 1900, **23**, 384.

Buttle, Hewitt, *J. Chem. Soc.*, 1909, **95**, 1759 (Footnote).

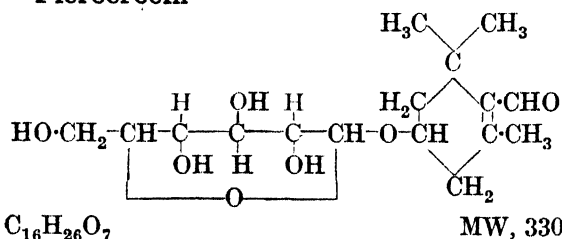
Jackson, Earle, *Am. Chem. J.*, 1903, **29**, 104.

Blanksma, *Chem. Zentr.*, 1909, **I**, 1809.

Marqueyrol, Scohy, *Bull. soc. chim.*, 1920, **27**, 105.

Guastalla, Racciu, *Industria chimica*, 1933, **8**, 1370.

### Picrocrocin



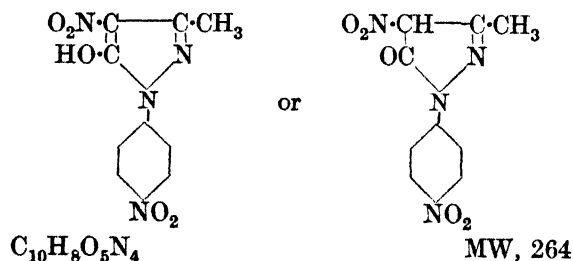
Terpene-glucoside, bitter principle from *Safran aquila* (*Safran electus*). Stout colourless prisms from  $MeOH-CHCl_3-Et_2O$ . M.p. 156°. Acid or alk. hyd.  $\rightarrow$  *d*-glucose + safranal.

*Tetra-acetate*: needles from pet. ether. M.p. 143°. *Semicarbazone*: m.p. 106°.

Kuhn, Winterstein, *Ber.*, 1934, **67**, 344.

Cf. Lutz, *Biochem. Z.*, 1930, **226**, 97.

### Picrolonic Acid (4-Nitro-3-methyl-1-p-nitrophenylpyrazolone-5)

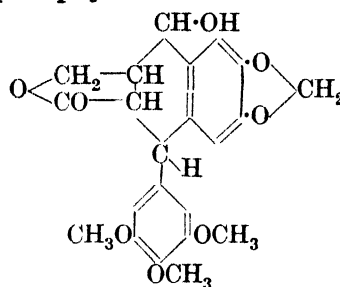


Yellow cryst. M.p. 116.5° (decomp. at 125°). Sol.  $H_2O$  to 0.12% at 17°. Very spar. sol.  $EtOH$ . Org. bases  $\rightarrow$  cryst. add. comps. of

definite m.p. Forms very insol. salts of Ca, Cu, Pb, hence used in detection of these metals.

Hugounenq, Florence, Couture, *Bull. soc. chim. biol.*, 1925, **7**, 58.

### Picropodophyllin

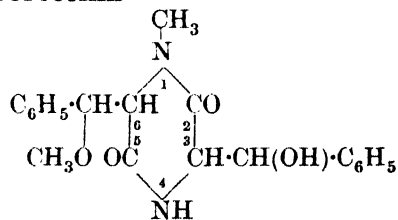


Constituent of purgative resin from various species of *Podophyllum*. Isomeric with podophyllotoxin but physiologically inactive. Colourless needles from  $MeOH$  or  $C_6H_6$ . Cryst. + 1 mol. solvent from  $MeOH$  or  $EtOH$ . M.p. 228°.  $[\alpha]_D^{20} + 9.38^\circ$  in  $CHCl_3$ . Fuming  $HCl$  at 110° in sealed tube  $\rightarrow$  pyromellitic acid. Alk.  $KMnO_4 \rightarrow$  gallic acid trimethyl ether + oxalic acid.  $Zn$  dust dist.  $\rightarrow$  1:6-dimethylnaphthalene.

*Acetyl*:  $C_{24}H_{24}O_9$ . MW, 456. M.p. 215-16°.  $[\alpha]_D^{18} + 17.7^\circ$  in  $CHCl_3$ .

Späth, Wesseley, Nadler, *Ber.*, 1933, **66**, 125.

### Picrorocellin



(An alternative structure has H at 1 and  $CH_3$  at 4)

$C_{20}H_{22}O_4N_2$  MW, 354

Constituent of lichen *Rocella fuciformis*. Massive prisms from boiling  $EtOH$ . M.p. 190-220° according to rate of heating (192-4°). Insol. cold dil. aq. acids and alkalis.  $[\alpha]_D^{18} + 12.5^\circ$  in  $CHCl_3$ . Heat alone or with boiling  $NaOH$ . Aq.  $\rightarrow$  anhydropicrorocellin.

O : N - Di - Me : dimethylpicrorocellin.  $C_{22}H_{26}O_4N_2$ . MW, 382. Prisms from boiling  $EtOH$ . M.p. 229°. Optically inactive. HI at 140°  $\rightarrow$  2:5-diketo-3:6-dibenzyl-1:6-dimethylpiperazine.

*Anhydropicrorocellin*:  $C_{20}H_{20}O_3N_2$ . MW, 336. Needles. M.p.  $155^\circ$ .  $[\alpha]_D^{18} - 463.7^\circ$  in  $CHCl_3$ . *Me ether*: prisms from  $EtOH-C_6H_6$ -pet. ether. M.p.  $139^\circ$ .  $[\alpha]_D^{18} - 661.2^\circ$  in  $CHCl_3$ .

Forster, Saville, *J. Chem. Soc.*, 1922, **121**, 816.

### Picrotin

$C_{15}H_{18}O_7$  MW, 310

Di-lactone constituent of the molecular comp. picrotoxin. Colourless rods from  $H_2O$ . M.p.  $255^\circ$  ( $252^\circ$ ).  $[\alpha]_D^{18} - 70^\circ$  in  $EtOH$ . Baryta-water or 0.1*N*-KOH in sealed tube  $\rightarrow$  acetone + a lactone  $C_{12}H_{14}O_2$ . Alk.  $KMnO_4 \rightarrow \alpha$ - and  $\beta$ -picrotinic acids.  $MnO_2 + H_2SO_4.Aq. \rightarrow$  1:1-dimethylphthalide-3:4-dicarboxylic acid.  $HCl$  at  $180^\circ \rightarrow$  a chloroketone,  $C_{14}H_{15}O_3Cl$ .

Mercer, Robertson, *J. Chem. Soc.*, 1936, 291; 1935, 997.

Clark, *J. Am. Chem. Soc.*, 1935, **57**, 1111.

Horrnann, *Ann.*, 1916, **411**, 273.

### $\alpha$ -Picrotinic Acid

$C_{14}H_{19}O_6.COOH$

$C_{15}H_{20}O_8$  MW, 328

Main product of prolonged hyd. of picrotin by dil. min. acids. Formed as ester together with K salt of  $\beta$ -picrotinic acid by action of  $MeOH-KOH$  on picrotin. Cryst. from  $AcOEt$ . M.p.  $258^\circ$  decomp.  $[\alpha]_D^{18} + 71^\circ 53'$  in  $EtOH$ . Very sol.  $H_2O$ ,  $EtOH$ ,  $MeOH$ ,  $Me_2CO$ ,  $AcOH$ . Insol.  $CHCl_3$ ,  $C_6H_6$ , ligroin. Heat in vacuo above m.p.  $\rightarrow$  picrotinlactone + picrotoxic acid.  $40\% H_2SO_4 \rightarrow$  picrotonol.

*Me ester*:  $C_{16}H_{22}O_8$ . MW, 342. Needles from  $H_2O$ . M.p.  $239^\circ$ .  $[\alpha]_D^{18} + 77^\circ 11'$  in  $EtOH$ .

*Et ester*:  $C_{17}H_{24}O_8$ . MW, 356. Needles from  $H_2O$ . M.p.  $199^\circ$ .  $[\alpha]_D^{18} + 74^\circ 25'$  in  $EtOH$ .

Horrnann, *Ann.*, 1916, **411**, 284, 298.

Horrnann, Seydel, *Ber.*, 1912, **45**, 3084.

### $\beta$ -Picrotinic Acid

$C_{14}H_{19}O_6.COOH$

$C_{15}H_{20}O_8$  MW, 328

Stout cryst. from  $H_2O$ . Decomp. at  $204-5^\circ$ . Sol.  $EtOH$ ,  $MeOH$ ,  $AcOH$ . Spar. sol.  $AcOEt$ ,  $Me_2CO$ . Insol.  $CHCl_3$ ,  $C_6H_6$ , ligroin.  $[\alpha]_D^{18} + 4^\circ 23'$  in  $EtOH$ . Hot  $KOH.Aq. \rightarrow$  picrotin-dicarboxylic acid.

*K salt*: cryst. from  $MeOH$ . Sinters at  $245-7^\circ$ , decomp. at  $260^\circ$ .  $[\alpha]_D^{18} - 3^\circ 57'$  in  $H_2O$ .

*Me ester*:  $C_{16}H_{22}O_8$ . MW, 342. M.p.  $231^\circ$ .

See previous references.

### Picrotoxic Acid

$C_{15}H_{18}O_7$  MW, 310

Needles +  $2H_2O$  from  $H_2O$ . M.p. anhyd.  $232^\circ$ . Very sol.  $MeOH$ ,  $EtOH$ ,  $AcOH$ . Sol.  $Me_2CO$ ,  $AcOEt$ ,  $H_2O$ . Insol.  $C_6H_6$ ,  $CHCl_3$ , ligroin.  $[\alpha]_D^{18} + 81^\circ 7'$  in  $EtOH$ . Decolourises bromine water. Reduces  $AgNO_3$  and Fehling's.  $H \rightarrow$  dihydro-acid.

*Me ester*:  $C_{16}H_{20}O_7$ . MW, 324. M.p.  $171.5^\circ$ .  $[\alpha]_D^{18} + 87^\circ 32'$  in  $EtOH$ .

*Et ester*:  $C_{17}H_{22}O_7$ . MW, 338. M.p.  $143.5^\circ$ .  $[\alpha]_D^{18} + 81^\circ 12'$  in  $EtOH$ .

Mercer, Robertson, *J. Chem. Soc.*, 1936, 293.

Horrnann, *Ber.*, 1916, **49**, 1554; *Ann.*, 1916, **411**, 300.

### Picrotoxin

$C_{30}H_{34}O_{13}$  MW, 602

Bitter principle from berries of shrubs of *Anamirta cocculus*, *Menispermum cocculus*, and other species. Is an equimolecular comp. of picrotin and picrotoxinin. Prisms from  $H_2O$  or  $EtOH$ . M.p.  $203-4^\circ$  ( $199-200^\circ$ ).  $[\alpha]_D^{12} - 30^\circ$  in  $H_2O$ .  $Br.Aq. \rightarrow$  monobromopicrotoxinin + picrotin.  $MnO_2 + H_2SO_4.Aq. \rightarrow$  1:1-dimethylphthalide-dicarboxylic acid.

Horrnann, Thilo, *Chem. Zentr.*, 1936, **I**, 2954.

Mercer, Robertson, *J. Chem. Soc.*, 1936, 291.

Clark, *J. Am. Chem. Soc.*, 1935, **57**, 1111.

Sielisch, *Ann.*, 1912, **391**, 1.

### $\alpha$ -Picrotoxinic Acid

$C_{14}H_{17}O_5.COOH$

$C_{15}H_{18}O_7$  MW, 310

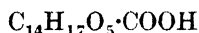
Lactone-acid. Cubes from  $H_2O$ . M.p.  $209^\circ$  decomp. Very sol.  $MeOH$ ,  $Me_2CO$ . Sol.  $EtOH$ ,  $AcOEt$ . Insol.  $C_6H_6$ ,  $CHCl_3$ .  $[\alpha]_D^{18} - 5^\circ$  in  $EtOH$ . Reduces cold  $KMnO_4.Aq.$ ,  $NH_3.AgNO_3$  and Fehling's on warming. Decolourises bromine water.  $H \rightarrow$  dihydro-deriv. Hot alkalis  $\rightarrow$  picrotoxinin-dicarboxylic acid. Refluxed 15 mins. with  $2N-H_2SO_4 \rightarrow \beta$ -picrotoxinic acid.

*Me ester*:  $C_{16}H_{20}O_7$ . MW, 324. Prisms from  $H_2O$ . M.p.  $182^\circ$ .  $[\alpha]_D^{18} - 9^\circ 44'$  in  $EtOH$ .

*Et ester*:  $C_{17}H_{22}O_7$ . MW, 338. Needles from  $H_2O$ . M.p.  $159^\circ$ .  $[\alpha]_D^{18} - 8^\circ 4'$  in  $EtOH$ .

Horrnann, *Ber.*, 1913, **46**, 2793.

Meyer, Bruger, *Ber.*, 1898, **31**, 2958.

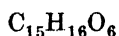
**$\beta$ -Picrotoxinic Acid** $C_{15}H_{18}O_7$  MW, 310

Needles from  $H_2O$ . M.p.  $235^\circ$  decomp. Very sol. MeOH, EtOH,  $Me_2CO$ , AcOH. Sol.  $Et_2O$ ,  $CHCl_3$ . Insol.  $C_6H_6$ , ligroin.  $[\alpha]_D^{18} - 48^\circ$  in EtOH. Stable in cold to Br.Aq. and  $KMnO_4$ .Aq. Non-reducing.

*Me ester*: prisms from EtOH. M.p.  $204^\circ$ .  $[\alpha]_D^{18} - 50.3^\circ$  in EtOH.

*Et ester*: prisms from  $H_2O$ . M.p.  $198^\circ$ .  $[\alpha]_D^{18} - 49.57^\circ$  in EtOH.

See previous references.

**Picrotoxinin**

MW, 292

Di-lactone constituent of the molecular comp. picrotoxin, of which it is the physiologically active (neurophilic) component. Colourless rods from  $H_2O$ . M.p.  $209.5^\circ$ . Reduces  $AgNO_3$  and Fehling's. Decolourises bromine water. HCl at  $180^\circ \rightarrow$  a chloroketone,  $C_{14}H_{15}O_5Cl$ . Heat with 10% alkali  $\rightarrow$  acetone + a lactone,  $C_{12}H_{14}O_2$ . Refluxed with 1%  $H_2SO_4$  for 24 hours  $\rightarrow$  picrotoxic acid.  $O_3$  in AcOEt  $\rightarrow$   $H \cdot CHO + \alpha$ -picrotoxinone.  $H(+Pd)$  in AcOEt  $\rightarrow$   $\beta$ -dihydropicrotoxinin, m.p.  $256-7^\circ$ .  $H(+Pt)$  in AcOH  $\rightarrow$   $\alpha$ -dihydro comp., m.p.  $252^\circ$ .

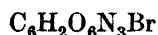
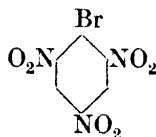
Mercer, Robertson, *J. Chem. Soc.*, 1936, 291; 1935, 997.

Clark, *J. Am. Chem. Soc.*, 1935, 57, 1111.

**Picrylaniline.**

See 2 : 4 : 6-Trinitrodiphenylamine.

**Picryl bromide** (2-Bromo-1 : 3 : 5-trinitrobenzene, 2 : 4 : 6-trinitrobromobenzene)

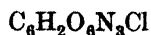
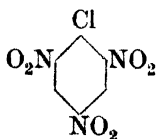


MW, 292

Yellowish plates from EtOH- $C_6H_6$ . M.p.  $122-3^\circ$ . Sol. EtOH,  $CHCl_3$ , AcOH,  $C_6H_6$ . Insol.  $H_2O$ . Hot NaOH.Aq.  $\rightarrow$  picric acid. NaOMe  $\rightarrow$  2 : 4 : 6-trinitroanisole.

Hertel, Römer, *Z. physik. Chem.*, 1933, 22B, 267.

**Picryl chloride** (2-Chloro-1 : 3 : 5-trinitrobenzene, 2 : 4 : 6-trinitrochlorobenzene)



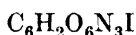
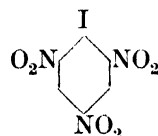
MW, 247.5

Needles or plates from  $CHCl_3$  or EtOH-ligroin. M.p.  $83^\circ$ .  $D^{20} 1.797$ . Very sol. hot  $CHCl_3$ ,  $C_6H_6$ . Sol. boiling EtOH. Spar. sol.  $Et_2O$ , hot ligroin. Insol.  $H_2O$ .  $Sn + HCl \rightarrow$  1 : 3 : 5-triaminobenzene.  $NH_3$ .Aq.  $\rightarrow$  2 : 4 : 6-trinitroaniline.

Desvergnes, *Chimie et Industrie*, 1931, 25, 3, 291

See also previous reference.

**Picryl iodide** (2-Iodo-1 : 3 : 5-trinitrobenzene, 2 : 4 : 6-trinitroiodobenzene)

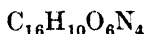
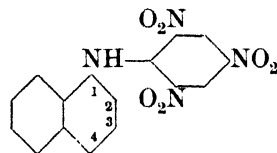


MW, 339

Golden-yellow tetragonal cryst. from  $C_6H_6$ . M.p.  $164-5^\circ$ .  $D^{25} 2.285$ . Hot NaOH.Aq.  $\rightarrow$  picric acid.

Hertel, Römer, *Z. physik. Chem.*, 1933, 22B, 267.

**N-Picryl-1-naphthylamine** (2 : 4 : 6-Trinitrophenyl-1-naphthylamine)



MW, 354

Red plates from AcOH, needles from EtOH. M.p.  $198-9^\circ$ . Insol. cold EtOH.

*N-Me*:  $C_{17}H_{12}O_6N_4$ . MW, 368. Plates from AcOH. M.p.  $247^\circ$  ( $245^\circ$ ). Sol. hot  $C_6H_6$ , AcOH. Spar. sol. hot EtOH.

Wedekind, *Ber.*, 1900, 33, 435.

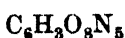
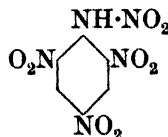
Busch, Kögel, *Ber.*, 1910, 43, 1560.

**N-Picryl-2-naphthylamine** (2 : 4 : 6-Trinitrophenyl-2-naphthylamine).

Red prisms from AcOH. M.p.  $233-233.5^\circ$ . Mod. sol. most org. solvents.

Bamberger, Müller, *Ber.*, 1900, 33, 107.

**Picrylnitramine** (2 : 4 : 6-Trinitrophenyl-nitramine, N : 2 : 4 : 6-tetranitroaniline)



MW, 273

Yellow cryst. from  $\text{Me}_2\text{CO}-\text{CHCl}_3$ . De-fragrates at  $80-110^\circ$ .

N-Me: see Tetryl.

N-Et: ethylpicrylnitramine.  $\text{C}_8\text{H}_7\text{O}_8\text{N}_5$ . MW, 301. Pale yellow plates from EtOH. M.p.  $96^\circ$ .  $\text{D}^{10}_{1.644}$ . Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Red.  $\rightarrow$  2:4:6-triaminophenol. Alkalis or  $\text{NH}_3 \rightarrow$  intense red col.

N-Propyl: propylpicrylnitramine.  $\text{C}_9\text{H}_9\text{O}_8\text{N}_5$ . MW, 315. Colourless plates from EtOH. M.p.  $98^\circ$ . Alkalis or  $\text{NH}_3 \rightarrow$  red col.

N-Isopropyl: isopropylpicrylnitramine. Pale yellow needles from EtOH. M.p.  $108^\circ$ . Sol. EtOH.  $\text{D}^{10}_{1.563}$ .

N-Butyl: butylpicrylnitramine.  $\text{C}_{10}\text{H}_{11}\text{O}_8\text{N}_5$ . MW, 329. Colourless plates from EtOH. M.p.  $98-9^\circ$ .

N-Isobutyl: isobutylpicrylnitramine. Needles from EtOH. M.p.  $110^\circ$ .

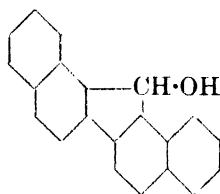
Jones, Willson, *J. Chem. Soc.*, 1930, 2277.

Duin, *Rec. trav. chim.*, 1917, **37**, 112.

Franchimont, *Rec. trav. chim.*, 1910, **29**, 300.

Romburgh, *Rec. trav. chim.*, 1885, **41**, 191.

### Picylene-carbinol



$\text{C}_{21}\text{H}_{14}\text{O}$

MW, 282

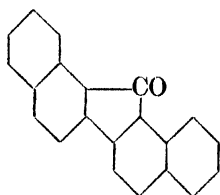
Pearly leaflets from  $\text{CHCl}_3$ . M.p.  $230^\circ$ . Sol. hot EtOH, AcOH,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ . Sol. in hot conc.  $\text{H}_2\text{SO}_4 \rightarrow$  blue col.

Acetate: needles. M.p.  $159^\circ$ .

Bamberger, Chattaway, *Ann.*, 1895, **284**, 69.

Cf. Schmidlin, Hüber, *Ber.*, 1910, **43**, 2824.

### Picylene-ketone



$\text{C}_{21}\text{H}_{12}\text{O}$

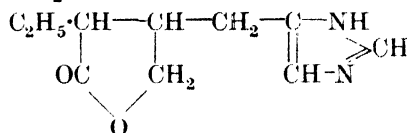
MW, 280

Golden-yellow cryst. from xylene. M.p.  $185.5^\circ$ . Sublimes on slow heating. Sol. AcOH

$\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , boiling EtOH. Sol. in conc.  $\text{H}_2\text{SO}_4 \rightarrow$  violet col.  $\text{NaHg} \rightarrow$  picylene-carbinol.

See first reference above.

### Pilocarpidine



$\text{C}_{10}\text{H}_{14}\text{O}_2\text{N}_2$

MW, 194

Alkaloid occurring in leaves of *Pilocarpus Jaborandi*, Holmes. Physiological action similar to pilocarpine, but weaker.

Natural product.

Syrup. Decomp. on dist. Sol.  $\text{H}_2\text{O}$ .  $[\alpha]^{20}_D + 81.3^\circ$  in  $\text{H}_2\text{O}$ ,  $+ 35.2^\circ$  in presence of alkali. Salts heated with alkali split off  $\text{NHMe}_2$ .  $\text{NaOEt} \rightarrow$  isopilocarpidine.  $\text{MeI} \rightarrow$  pilocarpine methiodide.

$B, \text{HCl}$ :  $[\alpha]^{20}_D + 72^\circ$  in  $\text{H}_2\text{O}$ .

$B, \text{HNO}_3$ : prisms from  $\text{H}_2\text{O}$ . M.p.  $137^\circ$ .  $[\alpha]^{20}_D + 73.2^\circ$  in  $\text{H}_2\text{O}$ . Mod. sol. EtOH.

$B, \text{HAuCl}_4$ : needles from AcOH. M.p.  $125^\circ$ .

$B, \text{H}_2\text{PtCl}_6$ : leaflets +  $4\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd.  $187^\circ$  decomp.

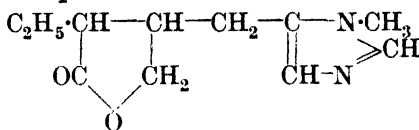
Synthetic product.

Racemic base: colourless cryst. M.p.  $128-9^\circ$ . Sol.  $\text{CHCl}_3$ .

Preobrashenski *et al.*, *Ber.*, 1936, **69**, 1837; 1933, **66**, 1537.

Späth, Kunz, *Ber.*, 1925, **58**, 513.

### Pilocarpine



$\text{C}_{11}\text{H}_{16}\text{O}_2\text{N}_2$

MW, 208

Alkaloid from leaves of various species of *Pilocarpus*. Colourless oil. B.p.  $260^\circ/5$  mm. (part. isomerisation).  $[\alpha]^{20}_D + 100.5^\circ$  (lowered in presence of alkali). Sol. alkalis  $\rightarrow$  unstable salts ("pilocarpates"). Heat alone or with alkali  $\rightarrow$  isopilocarpine.

$B, \text{HCl}$ : m.p.  $204-5^\circ$ .

$B, \text{HNO}_3$ :  $d$ -; m.p.  $178^\circ$ ; racemic, m.p.  $139-40^\circ$ .

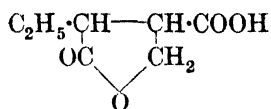
$B, \text{HAuCl}_4$ : m.p. anhyd.  $130^\circ$ .

Picrate: m.p.  $147^\circ$ .

Styphnate: m.p.  $183^\circ$ .

Preobrashenski *et al.*, *Ber.*, 1936, **69**, 1835; 1933, **66**, 1536, 1187; 1930, **63**, 460.

**Pilopic Acid** (*Ethylparaconic acid*, 2-keto-3-ethyltetrahydrofuran-4-carboxylic acid)



$C_7H_{10}O_4$

MW, 158

*d*-.  
M.p. 122°.  $[\alpha]_D^{18} + 54.6^\circ$  in  $H_2O$ .

*l*-.  
M.p. 122°.  $[\alpha]_D^{18} - 54^\circ$  in  $H_2O$ .

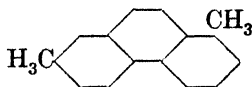
*dl*-.  
Needles from  $H_2O$ . M.p. 90–1° (86–7°).  
B.p. 210–15°/18 mm., 189–92°/7 mm. slight  
decomp. Sol. ord. org. solvents.

*Et ester*:  $C_9H_{14}O_4$ . MW, 186. Needles from  
EtOH.Aq. M.p. 49°. B.p. 283°/751 mm.  $D_4^{20}$   
1.1085. Sol. EtOH, Et<sub>2</sub>O. Insol.  $H_2O$ .

Preobrashenski *et al.*, *Ber.*, 1935, 68,  
847; 1930, 63, 460.

Welch, *J. Chem. Soc.*, 1931, 1370.

**Pimanthrene** (1 : 7-Dimethylphenanthrene)



$C_{16}H_{14}$

MW, 206

Plates from EtOH. M.p. 86°.

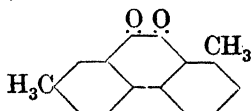
*Picrate*: needles from MeOH. M.p. 132°.

*Styphnate*: needles from MeOH. M.p. 159°.

Haworth, Letsky, Mavin, *J. Chem. Soc.*,  
1932, 1789.

Hosking, McFadyen, *J. Soc. Chem. Ind.*,  
1934, 53, 195T.

**Pimanthrenequinone** (1 : 7-Dimethylphen-  
anthraquinone)



$C_{16}H_{12}O_2$

MW, 236

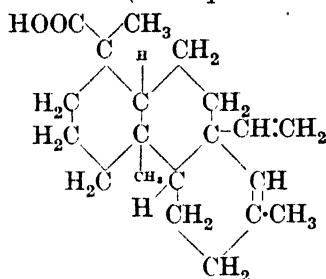
Plates from EtOH. M.p. 165°.

*Quinoxaline deriv.*: needles from  $CHCl_3$ -  
EtOH. M.p. 194–5°.

See first reference above and also

Ruzicka, Waldmann, *Helv. Chim. Acta*,  
1932, 15, 913.

***d*-Pimaric Acid** (*Dextropimaric acid*)



Suggested structure

$C_{26}H_{30}O_2$

MW, 302

Present in French colophony, etc. Prisms  
from EtOH. M.p. 218–19° (211°).  $[\alpha]_D^{20}$   
+ 87.3° in  $CHCl_3$ . Stable to heat and min.  
acids. Dehydrogenation  $\rightarrow$  pimanthrene.

*Me ester*:  $C_{21}H_{32}O_2$ . MW, 316. B.p. 149–  
50°/0.03 mm.  $D_4^{19}$  1.030.  $n_D^{19}$  1.52.

*Et ester*:  $C_{23}H_{34}O_2$ . MW, 330. B.p. 169–  
70°/0.2 mm.  $D_4^{14}$  1.013.  $n_D^{14}$  1.5151.

Palkin, Harris, *J. Am. Chem. Soc.*, 1933,  
55, 3677.

Ruzicka, Balas, *Helv. Chim. Acta*, 1923,  
6, 677.

Haworth, *J. Chem. Soc.*, 1932, 2717.

Ruzicka, de Graaff, Goldberg, Frank,  
*Helv. Chim. Acta*, 1932, 15, 915.

***l*-Pimaric Acid.**

*Note*.—Since the compound usually described  
by this name is not a stereoisomer of *d*-pimaric  
acid, it has recently been suggested that it  
should be renamed *l*-sapietic acid, *q.v.*

**Pimelic Acid** (*Pentane-1 : 5-dicarboxylic acid*)



$C_7H_{12}O_4$

MW, 160

Prisms from  $H_2O$ . M.p. 104–5°. B.p. 212°/  
10 mm. Sol.  $H_2O$ , EtOH, hot  $C_6H_6$ . Insol.  
cold  $C_6H_6$ . Non-volatile in steam.

*Me ester*:  $C_8H_{14}O_4$ . MW, 174. B.p. 181–2°/  
18 mm. *Chloride*:  $C_8H_{13}O_3Cl$ . MW, 192.5.  
B.p. 135–6°/17 mm.

*Di-Me ester*:  $C_9H_{16}O_4$ . MW, 188. B.p.  
130–5°/17 mm., 121–2°/11 mm.

*Et ester*:  $C_9H_{16}O_4$ . MW, 188. Cryst. from  
Et<sub>2</sub>O. M.p. 10°. B.p. 182°/18 mm., 162°/6  
mm.  $n_D^{20}$  1.4415. *Chloride*:  $C_9H_{15}O_3Cl$ . MW,  
206.5. B.p. 139°/17 mm.

*Di-Et ester*:  $C_{11}H_{20}O_4$ . MW, 216. B.p. 252–  
5°/748 mm., 153–6°/24 mm., 139–41°/15 mm.  
 $D_4^{20}$  0.99448.

*Diphenacyl ester*: cryst. from EtOH. M.p.  
72.4°.

*Di-p-bromophenacyl ester*: cryst. from EtOH. M.p. 136-6°.

*Dichloride*:  $C_7H_{10}O_2Cl_2$ . MW, 197. Oil. B.p. 137°/15 mm.

*Dinitrile*:  $C_7H_{10}N_2$ . MW, 122. B.p. 175-6°/14 mm.  $D^{18}_D$  0.949. Solidifies to a glass. Insol.  $H_2O$ .

*Anilide*: cryst. from  $H_2O$ . M.p. 108-9°.

*Dianilide*: cryst. from MeOH.Aq. M.p. 155-6°.

Meyer, *Helv. Chim. Acta*, 1933, **16**, 1292.

Müller, *Monatsh.*, 1934, **65**, 18.

Müller, Rölz, *Organic Syntheses*, 1931, XI, 42.

### Pimelic Dialdehyde (Heptandial-1 : 7)



$C_7H_{12}O_2$  MW, 128

Viscous oil with odour resembling tobacco. Very sol.  $H_2O$ . Readily polymerises to a white solid insol.  $H_2O$  or org. solvents.

*Dioxime*: cryst. from MeOH. M.p. 153°.

*Disemicarbazone*: cryst. from EtOH. M.p. 244° decomp.

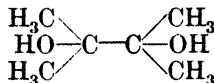
Weil, Traun, Marcel, *Ber.*, 1922, **55**, 2674.

Fischer, Düll, Ertel, *Ber.*, 1932, **65**, 1472.

### Pimelin-ketone.

See Cyclohexanone.

**Pinacol** (*Pinacone*, *dimethyl- $\psi$ -butylene glycol*, *tetramethylethylene glycol*, *2 : 3-dihydroxy-2 : 3-dimethylbutane*)



$C_6H_{14}O_2$  MW, 118

Cryst. from  $Et_2O$ , m.p. 38°: cryst. +  $6H_2O$  from  $H_2O$ , m.p. 47°. B.p. 175°. Sol. EtOH,  $Et_2O$ , hot  $H_2O$ . Spar. sol.  $CS_2$ , cold  $H_2O$ .

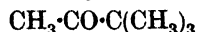
*Monoformyl*: b.p. about 90°/20 mm.

*Diacytyl*: m.p. 65°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

Adams, Adams, *Organic Syntheses*, Collective Vol. I, 448.

Friedel, Silva, *Ber.*, 1873, **6**, 267.

**Pinacolin** (*Pinacolone*, *methyl tert.-butyl ketone*, *1 : 1 : 1-trimethylacetone*)



$C_6H_{12}O$  MW, 100

B.p. 103-6°/746 mm.  $D^0$  0.8265,  $D^{18}_D$  0.7999.

*Oxime*: needles from EtOH.Aq. M.p. 74-5°.

B.p. 171.6°/748 mm.

*Phenylhydrazone*: oil. B.p. 165°/32 mm.

*2 : 4-Dinitrophenylhydrazone*: yellow cryst. from EtOH. M.p. 125°.

*Azine*: b.p. 213-16°, 103°/17 mm.

Hill, Flösdorf, *Organic Syntheses*, Collective Vol. I, 451.

Badertscher, Whitmore, *J. Am. Chem. Soc.*, 1932, **54**, 825.

### Pinacolin Alcohol.

See Methyl-tert.-butylcarbinol.

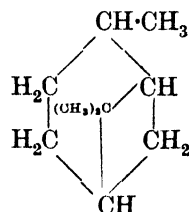
### Pinacolone.

See Pinacolin.

### Pinacone.

See Pinacol.

### Pinane



$C_{10}H_{18}$  MW, 138

Two stereoisomeric forms are possible but the homogeneity of any of the forms described has not been established.

*d.*-  $\alpha$ -Pinane.

B.p. 166-166.5°/762 mm.  $D^{20}_4$  0.8560.  $n^{20}_D$  1.4630.  $[\alpha]^{20}_D + 22.83^\circ$ .

*l.*-  $\beta$ -Pinane.

B.p. 167.5-168°/748 mm.  $D^{20}_4$  0.8567.  $n^{20}_D$  1.4605.  $[\alpha]^{20}_D - 19.84^\circ$ .

*dl.*- *Inactive* Pinane, pinocamphane.

B.p. 164.5-165°.  $D^{20}_4$  0.8551.  $n^{20}_D$  1.4609.

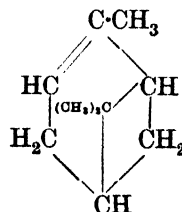
Rule, Chambers, *J. Chem. Soc.*, 1937, 151.

Lipp, *Ber.*, 1923, **56**, 2098.

Vavon, *Compt. rend.*, 1910, **150**, 1127

Nametkin, Jarzeff, *Ber.*, 1923, **56**, 833.

### $\alpha$ -Pinene



$C_{10}H_{16}$  MW, 136

Constituent of essential oils of Coniferae. Main constituent of oil of turpentine.

*d.*- *Australene*.

M.p. - 50°. B.p. 155-6°, 47-50°/10 mm.  $D^{20}_{15}$  0.862.  $n^{20}_D$  1.4685.  $[\alpha]^{20}_D + 48.3^\circ$ .

*Nitrosochloride*: m.p. 81-81.5°.  $[\alpha]^{20}_D + 322^\circ$ .

*l.* Firpene, terebenthene.

B.p. 155–6°.  $D^{20}_D$  0.8595.  $n^{20}_D$  1.47299.  $[\alpha]_D$  – 47.2°.

*Nitroschloride*: needles from EtOH–Et<sub>2</sub>O. M.p. 81–81.5°.  $[\alpha]_D$  – 322° in EtOH or CHCl<sub>3</sub>. *dl.*

Colourless oil. B.p. 156.2°.  $D^{20}_D$  0.8582.  $n^{20}_D$  1.4658. Heat at 250–70° → dipentene. Dry HCl → bornyl chloride.

*Nitroschloride*: leaflets from CHCl<sub>3</sub>–MeOH. M.p. 103°.

*Nitrosbromide*: cryst. from CHCl<sub>3</sub>–MeOH. M.p. 91–2° decomp.

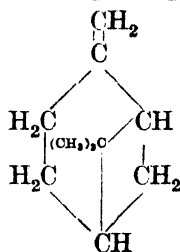
Lynn, *J. Am. Chem. Soc.*, 1919, **41**, 361.

Schorger, *J. Am. Chem. Soc.*, 1917, **39**, 1042.

Semmler, Bartelt, *Ber.*, 1907, **40**, 1368.

Wallach, *Ann.*, 1909, **368**, 2; 1890, **258**, 344.

$\beta$ -Pinene (*Nopinene*, *pseudopinene*)



$C_{10}H_{16}$

MW, 136

Constituent of oil of turpentine. B.p. 163–4°.  $D^{20}_D$  0.8675.  $n^{20}_D$  1.4749.  $[\alpha]_D$  – 22°. Dry HCl → bornyl chloride + dipentene dihydrochloride.

Vavon, *Compt. rend.*, 1910, **150**, 1129.

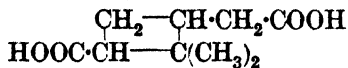
**Pinene Hydrate.**

See Methylnopinol.

“**Pinene hydrochloride.**”

See Bornyl chloride.

**Pinic Acid** (2:2-Dimethyl-3-carboxycyclobutylacetic acid, 2:2-dimethyl-3-carboxymethylcyclobutane-1-carboxylic acid)



$C_9H_{14}O_4$

MW, 186

*d.*

Prisms from Et<sub>2</sub>O–pet. ether. M.p. 135–6°. B.p. 212–16°/10 mm. Very sol. H<sub>2</sub>O, Et<sub>2</sub>O. Sol. Me<sub>2</sub>CO. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. pet. ether.  $[\alpha]_D$  + 7.1° in Me<sub>2</sub>CO.

*Di-Me ester*:  $C_{11}H_{18}O_4$ . MW, 214. Liq. B.p. 128–30°/9 mm.  $D^{20}_D$  1.0548.  $n^{20}_D$  1.4487.  $[\alpha]_D$  + 13.8°.

*Di-Et ester*:  $C_{13}H_{22}O_4$ . MW, 242. Liq. B.p. 142–6°/10 mm.  $D^{20}_D$  1.0104.  $n^{20}_D$  1.44962.  $[\alpha]_D$  + 8.0°.

*l.*

Needles from H<sub>2</sub>O. M.p. 135–6°.  $[\alpha]_D$  – 7.1° in Me<sub>2</sub>CO.

*dl.*

Prisms from H<sub>2</sub>O. M.p. 101–102.5°. B.p. 214–16°/9 mm. Spar. sol. cold H<sub>2</sub>O.

*Di-Me ester*: liq. B.p. 134–8°/17.5 mm.  $D^{20}_D$  1.053.  $n^{20}_D$  1.4490.

*Di-Et ester*: liq. B.p. 145–7°/10 mm.  $D^{20}_D$  1.0093.  $n^{20}_D$  1.44662.

Semmler, Mayer, *Ber.*, 1911, **44**, 3665.

Barbier, Grignard, *Bull. soc. chim.*, 1910, **7**, 548.

Baeyer, *Ber.*, 1896, **29**, 326.

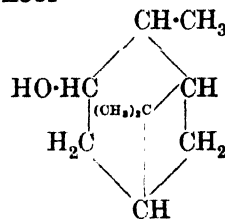
**Pinite.**

See under Inositol.

**Pinitol.**

See under Inositol.

**Pinocampheol**



$C_{10}H_{18}O$

MW, 154

*Cis*:

*d.*

M.p. 57°. B.p. 219°.  $[\alpha]_D$  + 37°.  $D^{15}_D$  0.973.

*Phthalate*: m.p. 126°.

*l.*

M.p. 57°. B.p. 219°.  $[\alpha]_D$  – 36°.  $D^{15}_D$  0.973.

*Phthalate*: m.p. 126°.

*Naphthylurethane*: m.p. 88°.

*dl.*

M.p. 42°. B.p. 219°.  $D^{15}_D$  0.973.

*Trans*:

*d.*

M.p. 67°. B.p. 217°.  $D^{15}_D$  0.968.  $n^{20}_D$  1.48330.  $[\alpha]_D$  + 55°.

*Phenylurethane*: m.p. 77°.

*l.*

M.p. 67°. B.p. 217°.  $D^{15}_D$  0.968.  $n^{20}_D$  1.48335.  $[\alpha]_D$  – 55°.

*Phthalate*: m.p. 107°.

*Phenylurethane*: m.p. 77°.

*Naphthylurethane*: m.p. 91°.

*dl.*

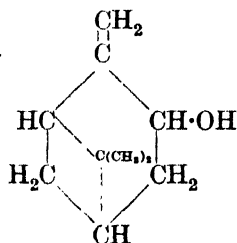
M.p. 36°. B.p. 217°.  $D^{15}_D$  0.968.

*Phthalate*: m.p. 113°.

*Phenylurethane*: m.p. 99°.

Schmidt, Schulz, *Chem. Zentr.*, 1934, II, 2077.

**Pinocarveol** (*Pinyl alcohol, isocarveol*)



$C_{10}H_{16}O$

MW, 152

*d.*

M.p. 7°. B.p. 208-9°/750 mm.  $D^{20}_D$  0.9815.  $n^{20}_D$  1.4993.  $[\alpha]_D + 59^\circ$ .

*Hydrate*: m.p. 190-1°.  $[\alpha]^{20}_D + 31^\circ$  in  $H_2O$ .

*Phenylurethane*: m.p. 88-9°.

*l.*

Occurs in oil from *Eucalyptus globulus*. M.p. 7°. B.p. 208-9°/750 mm.  $D^{20}_D$  0.981.  $n^{20}_D$  1.4996.  $[\alpha]_D - 62.19^\circ$ .

*Acetyl*: b.p. 227-8°.  $D^{20}_D$  0.997.  $[\alpha]_D + 15.8^\circ$ .

*1-Naphthylurethane*: m.p. 95°.

*dl.*

B.p. 215-18°.  $D^{22}_D$  0.978.  $n^{22}_D$  1.4979.  $H_2SO_4$  or  $KHSO_4 \rightarrow p$ -cymene.

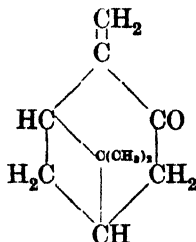
*Hydrate*: m.p. 176-7°.

*Phenylurethane*: m.p. 95-6°.

Schmidt, *Ber.*, 1929, 62, 2945; 1930, 63, 1129.

Wallach, *Ann.*, 1893, 277, 149; 1906, 346, 222.

**Pinocarvone** (*Isocarvone*)



$C_{10}H_{14}O$

MW, 150

*d.*

Oil. B.p. 222-3°.  $D^{20}_D$  0.9881.  $n^{20}_D$  1.50373.  $[\alpha]_D + 13^\circ$ .

*Oxime*: needles from MeOH.Aq. M.p. 68-9°.

*Semicarbazone*: needles. M.p. 212-15°.

*l.*

Oil. B.p. 222-3°.  $D^{20}_D$  0.987.  $n^{20}_D$  1.50390.  $[\alpha]_D - 15^\circ$ .

*Oxime*: needles from MeOH.Aq. M.p. 68-9°.

*Semicarbazone*: two forms. (i) Needles. M.p. 212-15°. (ii) M.p. 320°.

*dl.*

Oil with peppermint odour. B.p. 222-4°, 95°/12 mm.  $D^{19}_D$  0.989.  $n^{19}_D$  1.5067. Forms add. comp. with  $H_2S$ .  $NaHSO_3 \rightarrow$  unstable add. comp.

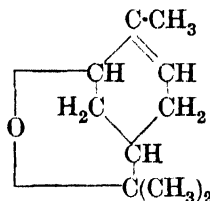
*Oxime*: m.p. 98°.

*Semicarbazone*: m.p. 204°.

Schmidt, *Ber.*, 1930, 63, 1131.

Wallach, *Ann.*, 1906, 346, 222.

**Pinol**



$C_{10}H_{16}O$

MW, 152

Liq. with odour resembling cineol. B.p. 183-4°, 76-7°/14 mm.  $D^{20}_D$  0.953.  $n^{20}_D$  1.4695.  $KMnO_4 \rightarrow$  terpenylic acid.

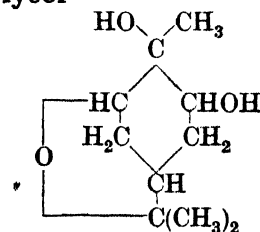
*Nitroschloride*: m.p. 103°.

Neave, *J. Chem. Soc.*, 1912, 101, 514.

Wallach, *Ann.*, 1896, 291, 349.

Wagner, *Ber.*, 1894, 27, 1644.

**Pinol Glycol**



$C_{10}H_{18}O_3$

MW, 186

*Cis*:

Needles from  $CHCl_3$ . M.p. 125°. B.p. 157°/12 mm.

*Di-Et ether*:  $C_{14}H_{26}O_3$ . MW, 242. Needles from  $Et_2O$ . M.p. 52-3°.

*Diacetyl*: needles from  $H_2O$ . M.p. 97-8°.

*Trans*:

Two forms:

(i) *Active*. M.p. 74°.

(ii) *Inactive*. Plates from  $H_2O$ . M.p. 128-9°. B.p. 157-8°/12 mm.

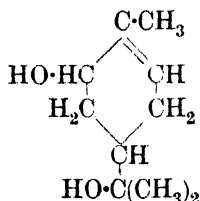


*Diacetyl*: m.p. 37–8°. B.p. 165–7°/17 mm.

Wallach, *Ann.*, 1892, **268**, 223; 1890, **259**, 311.

Wagner, Slawinski, *Ber.*, 1899, **32**, 2066; 1894, **27**, 1644.

**Pinol Hydrate** (dl- $\Delta^1$ -p-Menthenediol-6 : 8)



$C_{10}H_{18}O_2$

MW, 170

Plates or needles. M.p. 131°. B.p. 270–1°. Sol.  $H_2O$ , EtOH,  $Et_2O$ .  $H_2SO_4 \rightarrow$  red col.

*Diacetyl*: b.p. 159–161.5°.  $D_D^{18}$  1.0385.

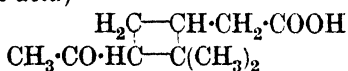
Wallach, *Ann.*, 1896, **291**, 351.

Henderson, Agnew, *J. Chem. Soc.*, 1909, **95**, 291.

### Pinonene.

See d- $\Delta^4$ -Carene.

**Pinonic Acid** (2 : 2-Dimethyl-3-acetocyclobutylacetic acid)



$C_{10}H_{16}O_3$

MW, 184

d-.

Cryst. from pet. ether. M.p. 69°. B.p. 168°/12 mm.  $[\alpha]_D^{25} + 88.27^\circ$  in  $CHCl_3$ .

*Oxime*: two forms. (i) Plates from MeOH. M.p. 129°. (ii) Cryst. from AcOH. M.p. 190–1°.

*Semicarbazone*: cryst. from EtOH. M.p. 204°.

l-.

Prisms from  $H_2O$  or  $C_6H_6$ -pet. ether. M.p. 68–9°.  $[\alpha]_D^{25} - 90.6^\circ$  in  $CHCl_3$ .

*Oxime*: two forms. (i) Plates from  $Et_2O$ -pet. ether. M.p. 129°. (ii) Cryst. from AcOH.Aq. M.p. 204°.

dl-.

Plates or prisms from MeOH. M.p. 103–5°. B.p. 180–7°/14 mm. Sol.  $CHCl_3$ .

*Oxime*: prisms from MeOH. M.p. 150°.

*Semicarbazone*: m.p. 206–7°.

*Me ester*:  $C_{11}H_{18}O_3$ . MW, 198. B.p. 254–5°/755 mm., 128°/11 mm.  $n_D^{17}$  1.4558.

*Note*.—Numerous oily forms are known which are mixtures of stereoisomers.

Schmidt, *Z. angew. Chem.*, 1929, **42**, 126.  
Ruzicka, Trebler, *Helv. Chim. Acta*, 1920, **3**, 762.

Barbier, Grignard, *Bull. soc. chim.*, 1910, **7**, 553.

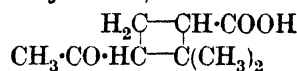
Tiemann, Semmler, *Ber.*, 1896, **29**, 532.

Perkin, Simonsen, *J. Chem. Soc.*, 1909, **95**, 1175.

Baeyer, *Ber.*, 1896, **29**, 23, 2786.

Tiemann, Kerschbaum, *Ber.*, 1900, **33**, 2664.

**Pinononic Acid** (2 : 2-Dimethyl-3-acetocyclobutane-1-carboxylic acid)



$C_9H_{14}O_3$

MW, 170

d-.

Prisms from  $H_2O$ . M.p. 129°.  $[\alpha]_D + 40.2^\circ$  in  $Et_2O$ .

*Semicarbazone*: m.p. 212° (204°).

l-.

Prisms from  $CHCl_3$ . M.p. 129°.

*Oxime*: prisms from  $Et_2O$ . M.p. 178–80°.

*Me ester*:  $C_{10}H_{16}O_3$ . MW, 184. B.p. 127°/13 mm.

Guha, Ganapathi, *Chem. Abstracts*, 1935, **29**, 5818.

Blumann, Zeitschel, *Ber.*, 1913, **46**, 1189.

Kerschbaum, *Ber.*, 1900, **33**, 891.

Wagner, Ertschikowsky, *Ber.*, 1896, **29**, 881.

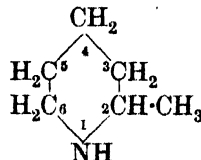
### $\alpha$ -Pipecolein.

See 6-Methyl-1 : 2 : 3 : 4-tetrahydropyridine.

### Pipelic Acid.

See Hexahydropicolinic Acid.

**$\alpha$ -Pipicoline** (2-Methylpiperidine, hexahydro- $\alpha$ -picoline)



$C_6H_{13}N$

MW, 99

d-.

B.p. 117–117.5°.  $[\alpha]_D^{15} + 18.7^\circ$  in  $CHCl_3$ .  $n_D^{15}$  1.44983.

*B.HCl*: m.p. 210°.

*B.HAuCl₄*: m.p. 131–2°.

*B₂H₂PtCl₆*: m.p. 194°.

*Picrate*: m.p. 116–17°.

d-Tartrate : m.p. anhyd. 110–12°.

l-Tartrate : m.p. anhyd. 126°.

N-Me :  $C_7H_{15}N$ . MW, 113. B.p. 127°.  $D^{16}_D$  0.825.  $[\alpha]^{15}_D + 68.8^\circ$ . Picrate : cryst. from MeOH. M.p. 240° decomp.

l-.

B,HCl : m.p. 190°.

B,HAuCl<sub>4</sub> : m.p. 131–2°.

B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub> : m.p. 194°.

Picrate : m.p. 116–17°.

d-Tartrate : m.p. anhyd. 126°.

l-Tartrate : m.p. anhyd. 111–12°.

N-Me :  $[\alpha]_D - 33.6^\circ$ .

dl-.

Liq. resembling piperidine in odour. B.p. 117–18°/747 mm.  $D^{23}_D$  0.8436.  $n^{23}_D$  1.4464. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

B,HCl : prisms from H<sub>2</sub>O. M.p. 210°.

B,HBr : needles. M.p. 189°.

B,HAuCl<sub>4</sub> : yellow prisms. M.p. 127° (118–19°).

B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub> : m.p. 202°.

Picrate : yellow needles. M.p. 134–5°.

N-p-Toluenesulphonyl : cryst. from ligroin. M.p. 54.5–55°.

N-Benzoyl : cryst. from EtOH. M.p. 44–5°.

N-Me : b.p. 126–7°/742 mm.  $D^{15}_D$  0.824.  $n^{14}_D$  1.4533. Sol. EtOH, Et<sub>2</sub>O, CS<sub>2</sub>. Sol. 10–12 parts cold H<sub>2</sub>O. Picrate : m.p. 240–1°. B,HCl : prisms from EtOH. M.p. 258–9°. B,HAuCl<sub>4</sub> : m.p. 215–16°. B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub> : m.p. 194–5°.

N-Et :  $C_8H_{17}N$ . MW, 127. B.p. 147–8°.  $D^{17}_D$  0.8368.  $n^{16}_D$  1.4480. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O. B,HAuCl<sub>4</sub> : plates from EtOH.Aq. M.p. 108°.

N-Propyl :  $C_9H_{19}N$ . MW, 141. B.p. 167–167.5°.  $D^{20}_D$  0.8296. Picrate : m.p. 113°.

N-Phenyl : b.p. 256.5–257°/710 mm. Picrate : prisms from H<sub>2</sub>O. M.p. 167–8°.

N-Benzyl : b.p. 267°, 160–2°/47 mm.

N-Nitroso : b.p. 123°/31 mm.

Borissow, Ber., 1930, 63, 2278.

Adkins, Kuick, Farlow, Wojcik, J. Am.

Chem. Soc., 1934, 56, 2425.

Skita, Brunner, Ber., 1916, 49, 1601.

Ladenburg, Ber., 1898, 31, 291.

Lipp, Ann., 1896, 289, 225.

Leithe, Ber., 1930, 63, 805.

**$\beta$ -Pipecoline** (3-Methylpiperidine, hexahydro- $\beta$ -picoline).

d-.

d-Tartrate : m.p. 76–8°.

l-Tartrate : m.p. 170°.

Dict. of Org. Comp.—III.

l-.

B.p. 124°.  $[\alpha]^{25}_D - 4^\circ$ .

d-Tartrate : needles. M.p. 170–2°.

dl-.

Liq. resembling piperidine in odour. B.p. 125–6°/763 mm.  $D^{24}_D$  0.8446.  $n^{24}_D$  1.4463. Very sol. H<sub>2</sub>O.

B,HCl : needles from C<sub>6</sub>H<sub>6</sub>. M.p. 171–2°.

B,HI : needles from C<sub>6</sub>H<sub>6</sub>. M.p. 158–9°.

B,HAuCl<sub>4</sub> : m.p. 130–1°.

B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub> : red prisms from H<sub>2</sub>O. M.p. 207°.

Picrate : yellow prisms. M.p. 136–8°.

d-Tartrate : m.p. 144–6°.

N-Me : oil. B.p. 124–6°.  $D^{15}_D$  0.818.

B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub> : orange prisms. M.p. 156–8°.

Methiodide : cryst. from EtOH. M.p. 196–7°.

N-Et : b.p. 145.5–146.5°.

N-p-Nitrophenyl : leaflets from EtOH. M.p. 61°.

N-2 : 4-Dinitrophenyl : yellow needles. M.p. 67°.

Franke, Kohn, Monatsh., 1879, 23, 878, 883.

Ladenburg, Heseckel, Ann., 1888, 247, 67.

Ladenburg, Ber., 1894, 27, 75.

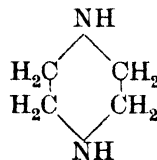
**$\gamma$ -Pipecoline** (4-Methylpiperidine, hexahydro- $\gamma$ -picoline).

Liq. which fumes in air. B.p. 126.5–129°.  $D^0$  0.8674. Sol. H<sub>2</sub>O.

Chloroaurate : yellow needles from H<sub>2</sub>O. M.p. 125–7°.

Ladenburg, Ann., 1888, 247, 69.

**Piperazine** (Diethylenediamine, hexahydropyrazine)



$C_4H_{10}N_2$

MW, 86

Hygroscopic plates from EtOH. M.p. 104°. B.p. 140°.

Hydrate : cryst. + 6H<sub>2</sub>O. M.p. 44°. B.p. 125–30°. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.  $k = 6.4 \times 10^{-5}$  at 25°. Aq. sol. reacts strongly alkaline. Dist. with Zn  $\rightarrow$  pyrazine.

B<sub>2</sub>C<sub>6</sub>H<sub>5</sub>OH : prisms from EtOH. M.p. 99–101°.

Picrate : yellow needles from H<sub>2</sub>O. M.p. 280° decomp.

N-Me :  $C_5H_{12}N_2$ . MW, 100. B.p. 134–6°.

$B, 2HCl, H_2O$ : cryst. M.p. 242–3°. *Dipicrate*: m.p. 272° decomp.

N:N-*Di-Me*:  $C_8H_{14}N_2$ . MW, 114. B.p. 131–2°.  $D_4^{20}$  0.8600.  $n_D^{20}$  1.4474.

N:N-*Di-Et*:  $C_8H_{18}N_2$ . MW, 142. B.p. 169–71° (165°).  $B, 2HCl$ : needles from EtOH. M.p. 277° decomp.

N:N-*Dipropyl*:  $C_{10}H_{22}N_2$ . MW, 170. B.p. 206°. *Dipicrate*: m.p. 258° decomp.

N-*Phenyl*: see N-Phenylpiperazine.

N:N-*Diphenyl*: see N:N-Diphenylpiperazine.

N:N-*Di-o-tolyl*:  $C_{18}H_{22}N_2$ . MW, 266. Needles from EtOH. M.p. 174°.

N:N-*Di-m-tolyl*: plates from EtOH. M.p. 126°.

N:N-*Di-p-tolyl*: prisms from pet. ether. M.p. 189–90°.

N:N-*Dibenzyl*:  $C_{18}H_{22}N_2$ . MW, 266. Needles from EtOH. M.p. 92°.

N-*Acetyl*: m.p. 52°.  $B, HCl$ : m.p. 181°.

N:N-*Diacetyl*: hygroscopic needles or plates from pet. ether. M.p. 144° (138°).

N-*Benzoyl*: m.p. 75°.  $B, HCl$ : m.p. 274°.

N:N-*Dibenzoyl*: m.p. 196°.

N:N-*Dibenzenesulphonyl*: cryst. from AcOH. M.p. 282–3°.

N-p-*Toluenesulphonyl*: m.p. 173° after sintering at 168°.

N:N-*Dinitroso*: yellow plates from  $H_2O$ . M.p. 158°.

Garelli, Racciu, *Angew. Chem.*, 1934, 47, 366.

Abderhalden, Klarmann, Schwab, *Z. physiol. Chem.*, 1924, 135, 180.

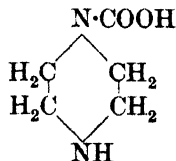
Pratt, Young, *J. Am. Chem. Soc.*, 1918, 40, 1429.

Prelog, Stepán, *Chem. Abstracts*, 1935, 29, 4013.

Jacobi, *Ber.*, 1933, 66, 113.

Abderhalden, Haas, *Z. physiol. Chem.*, 1925, 148, 245.

### Piperazine-N-carboxylic Acid



$C_5H_{10}O_2N_2$

MW, 130

Cryst. M.p. 162–5° (sealed tube).

*Et ester*:  $C_7H_{14}O_2N_2$ . MW, 158. B.p. 237°, 116–17°/12 mm. *Hydrochloride*: m.p. 145°.

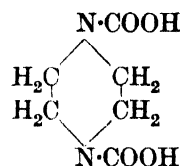
N'-*Benzoyl*: cryst. from ligroin. M.p. 82°.

N'-p-*Toluenesulphonyl*: cryst. from  $C_6H_6$ -ligroin. M.p. 121°.

Moore, Boyle, Thorn, *J. Chem. Soc.*, 1929, 39.

Rosdalsky, *J. prakt. Chem.*, 1896, 53, 24.

### Piperazine-N:N'-dicarboxylic Acid



$C_6H_{10}O_4N_2$

MW, 174

Free acid unknown.

*Di-Me ester*:  $C_8H_{14}O_4N_2$ . MW, 202. Cryst. from  $H_2O$ . M.p. 81°.

*Di-Et ester*:  $C_{10}H_{18}O_4N_2$ . MW, 230. Needles from ligroin. M.p. 45°.

*Diphenyl ester*:  $C_{18}H_{18}O_4N_2$ . MW, 326. Prisms. M.p. 177–8°.

*Di-1-naphthyl ester*:  $C_{26}H_{22}O_4N_2$ . MW, 426. M.p. 190–1°.

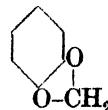
*Di-2-naphthyl ester*: m.p. 220°.

*Dinitrile*:  $C_6H_8N_4$ . MW, 136. Cryst. from EtOH. M.p. 168°.

van Dorp, *Rec. trav. chim.*, 1909, 28, 75.

Cazeneuve, Moreau, *Compt. rend.*, 1897, 125, 1183.

**Piperic Acid** (4-[3:4-Methylenedioxyphenyl]-1:4-butadiene-1-carboxylic acid, 3-piperonylideneacetic acid, 4-[3:4-methylenedioxyphenyl]-vinylacrylic acid)



$C_{12}H_{10}O_4$

MW, 218

Colourless needles from EtOH, turning yellow in light. M.p. 215°. Sol. hot EtOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ . Sublimes in yellow needles.  $H_2SO_4 \rightarrow$  bluish-violet col.  $KMnO_4$  at 3°  $\rightarrow$  piperonal.

*Me ester*:  $C_{13}H_{12}O_4$ . MW, 232. Yellow plates from MeOH. M.p. 146°.

*Et ester*:  $C_{14}H_{14}O_4$ . MW, 246. Plates from EtOH. M.p. 78°.

p-*Nitrobenzyl ester*: cryst. from 80% EtOH. M.p. 145°.

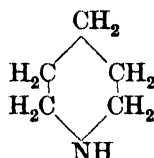
Ladenburg, Scholtz, *Ber.*, 1894, 27, 2959.

Babo, Keller, *J. prakt. Chem.*, 1857, 72, 56.

Fittig, Mielch, *Ann.*, 1869, 152, 27.

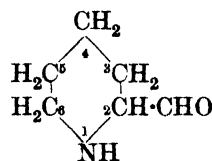
$\Delta^2$ -Piperideine.

See 1 : 2 : 3 : 4-Tetrahydropyridine.

**Piperidine** (*Hexahydropyridine*, *pentamethyleineimine*) $C_5H_{11}N$ 

MW, 85

Occurs in form of piperine in black pepper (*Piper nigrum*, *P. longum*, *P. officinarum*). Colourless liq. with characteristic odour. M.p.  $-9^\circ$ . B.p.  $106.0^\circ$ ,  $52.6^\circ/170$  mm.,  $17.7^\circ/20$  mm.  $D_4^{20}$  0.8606,  $D_4^{25}$  0.8336,  $D_4^{30}$  0.8033.  $n_D^{20}$  1.4530. Heat of comb.  $C_v$  825 Cal.  $k = 1.58 \times 10^{-3}$  at  $25^\circ$ . Misc. with  $H_2O$  in all proportions. Pyrolysis  $\rightarrow$  pyrrole. Forms a hydrate, m.p.  $-14^\circ$ . Forms complexes with heavy metal salts.

*B.HCl*: prisms from EtOH. M.p.  $244-5^\circ$ .*B.HBr*: prisms from  $CHCl_3$ . M.p.  $235^\circ$ .*B.HNO\_2*: plates. M.p.  $110^\circ$ .Acetate: m.p.  $106^\circ$ .*Picrate*: yellow needles from  $H_2O$ . M.p.  $151-2^\circ$ .*Styphnate*: m.p.  $231-2^\circ$ .*B.HAuCl\_4*: plates from EtOH. M.p.  $204^\circ$ .*N.Me*: see *N*-Methylpiperidine.*N.Et*: see *N*-Ethylpiperidine.*N-Propyl*:  $C_8H_{17}N$ . MW, 127. B.p.  $149-50^\circ$ . *Methiodide*: m.p.  $181-2^\circ$ . *Ethiodide*: m.p.  $276.5^\circ$ .*N-Isopropyl*: b.p.  $149-50^\circ$ .*N-Butyl*:  $C_9H_{19}N$ . MW, 141. B.p.  $175-6^\circ$ . *Methiodide*: m.p.  $198^\circ$ .*N-Amyl*:  $C_{10}H_{21}N$ . MW, 155. B.p.  $196^\circ$ . *Picrate*: m.p.  $107^\circ$ .*N-Isoamyl*: b.p.  $188^\circ$ . *Methiodide*: m.p.  $195^\circ$ .*N-Allyl*:  $C_8H_{15}N$ . MW, 125. B.p.  $151-2^\circ$ .  $D^{19}$  0.8445.*N-Benzyl*:  $C_{12}H_{17}N$ . MW, 175. B.p.  $245^\circ$ ,  $119^\circ/13$  mm.  $B_2H_2PtCl_6$ : m.p.  $191-3^\circ$ .*N-Phenyl*: see *N*-Phenylpiperidine.*N-Picryl*: yellow prisms from EtOH. M.p.  $104-6^\circ$ .*N-Triphenylmethyl*: needles from EtOH. M.p.  $153^\circ$ .*N-Formyl*: piperidine-*N*-aldehyde.  $C_6H_{11}ON$ . MW, 113. B.p.  $222^\circ$ . *B.HBr*: m.p.  $103.5^\circ$ .*N-Acetyl*: b.p.  $226-7^\circ$ .  $B_2H_2PtCl_6$ : red cryst. M.p.  $107-9^\circ$ .*N-Benzoyl*: m.p.  $48^\circ$ . B.p.  $320-1^\circ$ ,  $108-4^\circ/20$  mm.*N-p-Bromobenzoyl*: plates from EtOH. M.p.  $95^\circ$ .*N-p-Nitrobenzoyl*: yellow cryst. from EtOH. M.p.  $120.5^\circ$ .*N-3:5-Dinitrobenzoyl*: cryst. from EtOH. M.p.  $147^\circ$ .*N-Benzenesulphonyl*: prisms. M.p.  $93-4^\circ$ .*N-p-Toluenesulphonyl*: m.p.  $95-6^\circ$  ( $103^\circ$ ).*N-Nitroso*: yellow oil. B.p.  $218^\circ$ ,  $109^\circ/20$  mm.Marvel, Lazier, *Organic Syntheses*, Collective Vol. I, 93.Skita, Brunner, *Ber.*, 1916, **49**, 1600.Babo, Keller, *J. prakt. Chem.*, 1857, **72**, 55.Marie, Lejeune, *J. chim. phys.*, 1925, **22**, 59.Skita, Meyer, *Ber.*, 1912, **45**, 3592.Wojcik, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 2419.Staudinger, Müller, *Ber.*, 1923, **56**, 711.Ouchakof, Lifschitz, Jdanova, *Bull. soc. chim.*, 1935, **2**, 573.**Piperidine-2-aldehyde** (*2-Formylpiperidine*, *2-aldehydopiperidine*) $C_6H_{11}ON$ 

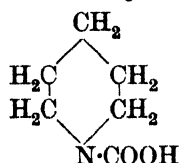
MW, 113

*Di-Et acetal*:  $C_{10}H_{21}O_2N$ . MW, 187. B.p.  $95-105^\circ/14$  mm.*p-Nitrophenylhydrazone hydrochloride*: yellow cryst. M.p.  $228^\circ$  decomp.Harries, Lénart, *Ann.*, 1915, **410**, 105.**Piperidine-3-aldehyde** (*3-Formylpiperidine*, *3-aldehydopiperidine*).*N-Et*:  $C_8H_{15}ON$ . MW, 141. B.p.  $40^\circ/0.2$  mm. *Di-Et acetal*:  $C_{12}H_{25}O_2N$ . MW, 215. B.p.  $63-5^\circ/0.04$  mm.*Di-Et acetal*: b.p.  $104^\circ/9$  mm.*m-Nitrophenylhydrazone hydrochloride*: yellow cryst. M.p.  $232-3^\circ$  decomp.Wohl, Losanitsch, *Ber.*, 1907, **40**, 4695.**Piperidine-N-aldehyde.**

See under Piperidine.

**Piperidine-carboxylic Acid.**

See Hexahydroisonicotinic Acid, Hexahydro-nicotinic Acid, and Hexahydro-picolinic Acid.

Piperidine-*N*-carboxylic Acid $C_6H_{11}O_2N$ 

MW, 129

Free acid unknown.

*Me ester*:  $C_7H_{13}O_2N$ . MW, 143. B.p. 201°.*Et ester*: piperidylurethane, pentamethylenurethane.  $C_8H_{15}O_2N$ . MW, 157. B.p. 211–12°, 103°/20 mm.*Phenyl ester*:  $C_{13}H_{15}O_2N$ . MW, 205. Plates from EtOH. M.p. 80°.*2-Naphthyl ester*:  $C_{16}H_{17}O_2N$ . MW, 255. Needles from EtOH. M.p. 107°.*Chloride*:  $C_6H_{10}ONCl$ . MW, 147.5. B.p. 237–8°, 112°/13 mm.*Amide*: piperidylurea.  $C_6H_{12}ON_2$ . MW, 128. Needles from EtOH. M.p. 105–6°.*Nitrile*: *N*-cyanopiperidine.  $C_6H_{10}N_2$ . MW, 110. B.p. 124°/30 mm., 102°/10 mm.Schotten, *Ber.*, 1883, 16, 647.Bouchetal, la Roche, *Bull. soc. chim.*, 1903, 29, 753; 1902, 27, 451.

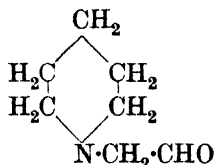
## Piperidine-3 : 4-dicarboxylic Acid.

See Hexahydrocinchomeronic Acid.

## Piperidinic Acid.

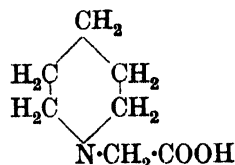
See 3-Amino-*n*-butyric Acid.

## Piperidinoacetaldehyde

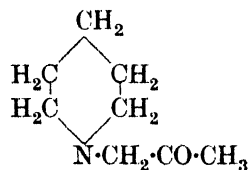
 $C_7H_{13}ON$ 

MW, 127

Not known in free state.

*B, HCl*: cryst. from Et<sub>2</sub>O. M.p. 103°. De-comp. on standing in air.*Chloroaurate*: yellow cryst. M.p. 109–11°.*Chloroplatinate*: orange-yellow needles. M.p. 121–2°.*Oxime*: cryst. from EtOH–Et<sub>2</sub>O. M.p. 135–6°.*Semicarbazone*: cryst. M.p. 76°.*Acetal*:  $C_{11}H_{23}O_2N$ . MW, 201. Liq. B.p. 219–21°. *B, H, AuCl<sub>4</sub>*: yellow plates. M.p. 96°.*B<sub>2</sub>, H<sub>2</sub>, PtCl<sub>6</sub>*: orange needles. M.p. 134°. *Methiodide*: cryst. M.p. 121°. *Ethiodide*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 123° (105°). *Picrate*: yellow needles. M.p. 67°.Stoermer, *Ber.*, 1898, 31, 2542.Stoermer, Burkert, *Ber.*, 1894, 27, 2016.Piperidinoacetic Acid (*Piperidine-1-acetic acid, pentamethyleneglycine*) $C_7H_{13}O_2N$ 

MW, 143

Prisms + 1H<sub>2</sub>O from EtOH. M.p. anhyd. 215–17°. Sol. H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, ligroin, C<sub>6</sub>H<sub>6</sub>. Sublimes.*B, HCl*: leaflets from EtOH–Et<sub>2</sub>O. M.p. 215–16°.*Methochloride*: m.p. 213° decomp.*Methochloroaurate*: needles from H<sub>2</sub>O. M.p. 178–9°.*Methochloroplatinate*: orange cryst. M.p. 219°.*Me ester*:  $C_8H_{15}O_2N$ . MW, 157. Oil. B.p. 205–7°.*Et ester*:  $C_9H_{17}O_2N$ . MW, 171. Oil. B.p. 209°/732 mm. *B, HCl*: m.p. 130–1°. *Methochloride*: m.p. 189°. Hygroscopic. *Methiodide*: cryst. from EtOH. M.p. 158–9°. *Methochloroplatinate*: orange cryst. M.p. 225°.*Nitrile*:  $C_7H_{12}N_2$ . MW, 124. Cryst. M.p. 19°. B.p. 210°, 99–100°/15 mm. *Methiodide*: leaflets. M.p. 192–3°.Ley, *Ber.*, 1909, 42, 367.Wedekind, *Ber.*, 1902, 35, 182; 1899, 32, 724.Bischoff, *Ber.*, 1898, 31, 2840.Kraut, *Ann.*, 1871, 157, 66.Piperidinoacetone (*N*-Acetonylpiperidine) $C_8H_{15}ON$ 

MW, 141

Liq. B.p. 195–7°. Misc. with H<sub>2</sub>O and common org. solvents.*B, H, AuCl<sub>4</sub>*: yellow plates. M.p. 107–8°.*B<sub>2</sub>, H<sub>2</sub>, PtCl<sub>6</sub>*: orange prisms. M.p. 192–3°.*Methiodide*: cryst. from EtOH. M.p. 126°.*Methochloroaurate*: cryst. M.p. 85°.*Methochloroplatinate*: orange cryst. from EtOH. M.p. 218–19°.*Oxime*: needles from H<sub>2</sub>O, plates from pet. ether. M.p. 104–5° (123°).

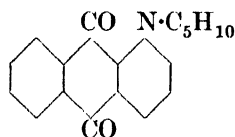
*Phenylhydrazone*: yellow leaflets from EtOH.Aq. M.p. 59–62°.

*2:4-Dinitrophenylhydrazone*: yellow needles from MeOH. M.p. 120–2°.

Matthaiopoulos, *Ber.*, 1898, **31**, 2398.

Stoermer, Burkert, *Ber.*, 1895, **28**, 1250.

### 1-Piperidinoanthraquinone



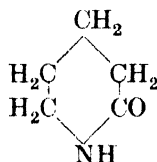
$C_{19}H_{17}O_2N$

MW, 291

Orange leaflets. M.p. 115°. Sol. conc.  $H_2SO_4 \rightarrow$  yellow sol. Sol.  $CHCl_3$ -AcOH  $\rightarrow$  purple sol.

Bayer, D.R.P., 136,777, (*Chem. Zentr.*, 1902, II, 1372).

### $\alpha$ -Piperidone (*Valerolactam*, 2-ketopiperidine)



$C_5H_9ON$

MW, 99

Hygroscopic cryst. M.p. 39–40°. B.p. 256°, 137°/14 mm., 64–5°/0.4 mm. Sol. EtOH, Et<sub>2</sub>O, H<sub>2</sub>O. Sol. dil. min. acids. Insol. alkalis.

*B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 182–3°.

*B.HgCl<sub>2</sub>*: needles + 1H<sub>2</sub>O. M.p. 187° decomp.

*N-Me*:  $C_6H_{11}ON$ . MW, 113. Hygroscopic liq. B.p. 104°/14 mm. *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 104°. *B.HgCl<sub>2</sub>*: needles + 1H<sub>2</sub>O. M.p. 119–20°.

*N-Et*:  $C_7H_{13}ON$ . MW, 127. Liq. B.p. 109°/12 mm. *B.HCl*: hygroscopic needles from EtOH-Et<sub>2</sub>O. M.p. 108°. *B.HgCl<sub>2</sub>*: needles + 1H<sub>2</sub>O. M.p. 113°.

*N-Propyl*:  $C_8H_{15}ON$ . MW, 141. Liq. B.p. 121°/14 mm. *B.HCl*: m.p. 112°.

*N-Isopropyl*: liq. B.p. 127–8°/15 mm. *B.HCl*: needles from EtOH-Et<sub>2</sub>O. M.p. 118°. *B.HgCl<sub>2</sub>*: m.p. 140–1°.

*N-Butyl*:  $C_9H_{17}ON$ . MW, 155. Liq. B.p. 130–1°/11 mm.

*N-Octyl*:  $C_{13}H_{25}ON$ . MW, 211. Liq. B.p. 172°/10 mm.

*N-Benzyl*:  $C_{12}H_{15}ON$ . MW, 189. Liq. B.p. 193°/8 mm.

*N-Acetyl*: liq. B.p. 238°.

*N-Benzoyl*: leaflets from EtOH. M.p. 112°.

*N-m-Nitrobenzoyl*: yellow leaflets from EtOH.Aq. M.p. 114°.

Räth, *Ann.*, 1931, **489**, 111.

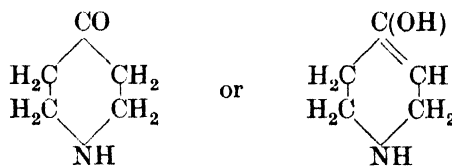
Ruzicka, *Helv. Chim. Acta*, 1921, **4**, 474.

Fischer, Zemplén, *Ber.*, 1909, **42**, 4886.

Fischer, Bergmann, *Ann.*, 1913, **398**, 114.

Wallach, *Ann.*, 1900, **312**, 179; 1902, **324**, 285.

### $\gamma$ -Piperidone (4-Ketopiperidine)



$C_5H_9ON$

MW, 99

Yellow oil which cannot be distilled. Heat.  $\rightarrow$  condensation products.

*B.HCl*: cryst. + 1½EtOH from EtOH-Et<sub>2</sub>O. M.p. 139–41°. M.p. (solvent free) 147–9°. Cryst. + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 94–6°.

*N-Me*:  $C_6H_{11}ON$ . MW, 113. *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 94–5°.

*N-Et*:  $C_7H_{13}ON$ . MW, 127. *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 105–6°.

*N-Propyl*:  $C_8H_{15}ON$ . MW, 141. *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 117–18°.

*N-Butyl*:  $C_9H_{17}ON$ . MW, 155. *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 178–80°.

*N-Phenyl*:  $C_{11}H_{13}ON$ . MW, 175. *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 145–7°.

*N-Benzyl*:  $C_{12}H_{15}ON$ . MW, 189. *B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 159–61°.

*O-Me*: b.p. 190–5–191°/738 mm. Volatile in steam. Strongly alkaline. *B.HgCl<sub>2</sub>*: needles from H<sub>2</sub>O. M.p. 191°.

*O-Et*: b.p. 96°/15 mm. Misc. with EtOH. Insol. H<sub>2</sub>O.

*O-Propyl*: b.p. 218–20°/742 mm. *B.HCl*: needles from EtOH-Et<sub>2</sub>O. M.p. 156–7°.

*Picrate*: prisms from EtOH. M.p. 120–4°.

Kuettel, McElvain, *J. Am. Chem. Soc.*, 1931, **53**, 2696.

Bolyard, *J. Am. Chem. Soc.*, 1930, **52**, 1032.

McElvain, Bolyard, *J. Am. Chem. Soc.*, 1929, **51**, 924.

Ruzicka, Fornasir, *Helv. Chim. Acta*, 1920, **3**, 806.

Koenigs, Neumann, *Ber.*, 1915, **48**, 960.



*d*.-

Liq. B.p. 165–70°/200 mm.  $D_{30}^{20}$  0.911.  $n_D^{20}$  1.474.  $[\alpha]_D + 46.0^\circ$ .

*l*.-

Liq. B.p. 95–6°/10 mm.  $D^{22}$  0.923.  $n_D^{22}$  1.476.  $[\alpha]_D - 34.1^\circ$ .

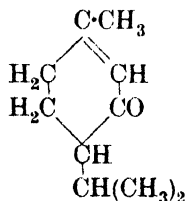
*dl*.-

Viscous oil with pleasant odour. B.p. 100–6°/19.5 mm.  $n_D^{18}$  1.477.

Read, Walker, *J. Chem. Soc.*, 1934, 308.

Read, Storey, *J. Chem. Soc.*, 1930, 2772, 2779.

**Piperitone** ( $\Delta^1$ -*p*-Menthenone-3, 1-methyl-4-isopropylcyclohexenone-3)

 $C_{10}H_{16}O$ 

MW, 152

*d*.-

Constituent of Japanese peppermint oil, essential oil from *Andropogon Jwarancusa* and *Cymbopogon sennarensis*. Colourless oil with camphoraceous odour. Turns yellow on standing in air. B.p. 116–118.5°/20 mm.  $D_4^{20}$  0.9344.  $n_D^{20}$  1.4848.  $[\alpha]_D^{20} + 49.13^\circ$ .

Semicarbazone: m.p. 193–4°.  $[\alpha]_D - 216.8^\circ$ .

*l*.-

Occurs in essential oil of *Eucalyptus Dives*. B.p. 109.5–110.5°/15 mm.  $D_4^{20}$  0.9324.  $n_D^{20}$  1.4848.  $[\alpha]_D^{20} - 51.53^\circ$ .

Oxime: oil.  $[\alpha]_D + 238.1^\circ$ .

*dl*.-

Constituent of *Eucalyptus Dives* oil. B.p. 232–3°/769 mm., 113°/18 mm.  $D_4^{20}$  0.9331.  $n_D^{20}$  1.4845.

Oxime: two forms. (i) Cryst. from EtOH. M.p. 118–19°. (ii) M.p. 88–9°.

Semicarbazone: two forms. (i) M.p. 224–6°. (ii) M.p. 171–2°.

Walker, *J. Chem. Soc.*, 1935, 1585.

Read, Walker, *J. Chem. Soc.*, 1934, 308.

Howard, E.P., 410,813, (*Chem. Abstracts*, 1934, 6446).

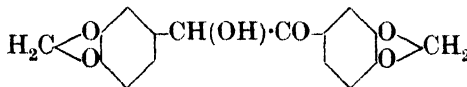
Stephan, Düker, *J. prakt. Chem.*, 1931, 129, 145.

Read, Smith, *J. Chem. Soc.*, 1923, 123, 2268.

Simonsen, *J. Chem. Soc.*, 1921, 119, 1646.

**Piperoin**

(Piperonyloin, piperonoin, 3 : 4 : 3' : 4'-dimethylenedioxybenzoin)

 $C_{16}H_{12}O_6$ 

MW, 300

Needles from EtOH. M.p. 120°. Sol. EtOH,  $CHCl_3$ . Spar. sol.  $Et_2O$ . Fehling's  $\rightarrow$  piperil.

Benzoyl: prisms from AcOH. M.p. 169°.

Oxalyl: crystals from  $PhNO_2$ . M.p. 169°.

Greene, Robinson, *J. Chem. Soc.*, 1922, 121, 2187.

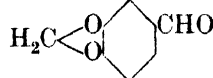
Smith, *Ann.*, 1896, 289, 324.

Perkin, *J. Chem. Soc.*, 1891, 59, 164.

**2-Piperolidine.**

See  $\delta$ -Coniceine.

**Piperonal** (Heliotropin, piperonyl aldehyde, 3 : 4-methylenedioxybenzaldehyde, methylene ether of protocatechuic aldehyde)

 $C_8H_6O_3$ 

MW, 150

Occurs in *Robinia pseudoacacia*. Cryst. from  $H_2O$ . M.p. 37°. B.p. 263°, 185°/78 mm., 140°/15 mm.

$2C_8H_6O_3, 3H_2SO_4$ : plates. M.p. 70–9°.

$2C_8H_6O_3, SnCl_4$ : yellow cryst. powder. Decomp. about 130°.

$2C_8H_6O_3, SnBr_4$ : m.p. 150°.

$C_8H_3(NO_3)_2 \cdot 1 : 3 : 5$  add. comp.: golden-yellow plates. M.p. 79°.

Oxime: *syn*-, cryst. from MeOH. M.p. 146°.

Acetyl: m.p. 99°. Benzoyl: m.p. 168°. Anti-, needles from  $H_2O$ . M.p. 112°. Acetyl: m.p. 86°.

Di-Me acetal:  $C_{10}H_{12}O_4$ . MW, 196. Liq. B.p. 267–9°.  $D^{15}$  1.206.

Di-Et acetal:  $C_{12}H_{16}O_4$ . MW, 224. Liq. B.p. 279–81°, 153–4°/11 mm.  $D^{15}$  1.129.

Imide:  $C_8H_6O_2N$ . MW, 149.  $B, HCl$ : needles from  $C_6H_6$ . M.p. about 229–30°.

Methylimide:  $C_9H_9O_2N$ . MW, 163. M.p. 46°. B.p. 148°/16 mm.  $B, HCl$ : m.p. 220°.

Cyanhydrin: see under 3 : 4-Methylenedioxy-mandelic Acid.

Anil: piperonylideneaniline.  $C_{14}H_{10}O_2N$ . MW, 225. Needles from ligroin. M.p. 65°.

Diacetyl: cryst. from EtOH. M.p. 80° (51°).

Semicarbazone: leaflets. M.p. 230–3°.

Phenylhydrazone: needles from EtOH. M.p. 106°.

*p*-Bromophenylhydrazone: leaflets from EtOH, AcOH or  $C_6H_6$ . M.p. 155° decomp.



*p*-Nitrophenylhydrazone: red cryst. M.p. 199–200°.

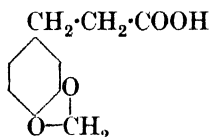
2 : 4-Dinitrophenylhydrazone: purplish-red cryst. M.p. 265°.

McLang, *Chem. Abstracts*, 1927, **21**, 77.

Fritzsche, D.R.P., 207,702, (*Chem. Zentr.*, 1909, I, 1207).

Ciamician, Silber, *Ber.*, 1890, **23**, 1160.

**Piperonylacetic Acid** (3 : 4-Methylenedioxy-hydrocinnamic acid)



$\text{C}_{10}\text{H}_{10}\text{O}_4$  MW, 194

Needles from  $\text{H}_2\text{O}$ . M.p. 87–8°. B.p. 171–2°/11–12 mm. Red sol. in  $\text{H}_2\text{SO}_4$ .  $\text{P}_2\text{O}_5 \rightarrow$  methylenedioxyhydrindone.

*Et ester*:  $\text{C}_{12}\text{H}_{14}\text{O}_4$ . MW, 222. Liq. B.p. 303°.

*Chloride*:  $\text{C}_{10}\text{H}_9\text{O}_3\text{Cl}$ . MW, 212.5. Yellow oil. Dist. under diminished pressure  $\rightarrow$  5 : 6-methylenedioxyhydrindone.

*Amide*:  $\text{C}_{10}\text{H}_{11}\text{O}_3\text{N}$ . MW, 193. Cryst. from  $\text{H}_2\text{O}$  or ligroin. M.p. 123–5°.

*Methylamide*:  $\text{C}_{11}\text{H}_{13}\text{O}_3\text{N}$ . MW, 207. Needles from  $\text{Et}_2\text{O}$ . M.p. 134°.

*Anilide*: needles. M.p. 122–3°.

Perkin, Robinson, *J. Chem. Soc.*, 1907, **91**, 1084.

Lorenz, *Ber.*, 1880, **13**, 758.

Regel, *Ber.*, 1887, **20**, 419.

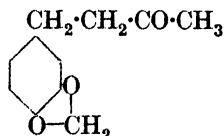
Piccinini, *Chem. Zentr.*, 1904, I, 879.

Brochet, Bauer, *Bull. soc. chim.*, 1915, **17**, 52.

Decker, *Ann.*, 1913, **395**, 289.

Borsche, Eberlein, *Ber.*, 1914, **47**, 1470.

**Piperonylacetone** (3 : 4-Methylenedioxybenzylacetone, methyl 3 : 4-methylenedioxyphenylethyl ketone, 3 : 4-methylenedioxy-1- $\gamma$ -ketobutylbenzene)



$\text{C}_{11}\text{H}_{12}\text{O}_3$  MW, 192

Leaflets from EtOH or pet. ether. M.p. 55°. B.p. 176–5°/15 mm., 164–5°/12 mm. Sol. most org. solvents.

*Oxime*: cryst. from EtOH or  $\text{Et}_2\text{O}$ . M.p. 98°.

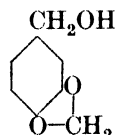
*Semicarbazone*: needles. M.p. 166°.

Vavon, Faillebin, *Compt. rend.*, 1919, **169**, 66.

Kaufmann, Radosević, *Ber.*, 1916, **49**, 679.

Brochet, Cabaret, *Compt. rend.*, 1914, **159**, 328.

**Piperonyl Alcohol** (3 : 4-Methylenedioxybenzyl alcohol)



$\text{C}_8\text{H}_8\text{O}_3$  MW, 152

Needles from pet. ether. M.p. 58°. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Dist.  $\rightarrow$  piperonal. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  2 : 3 : 6 : 7-dimethylenedioxy-9 : 10-dihydroanthracene.

*Acetyl*: two forms. (i) Cryst. from  $\text{CCl}_4$  or pet. ether. M.p. 51°. (ii) Liq. B.p. 153–4°/14 mm.  $D_4^{18}$  1.240.  $n_D^{18}$  1.528.

*Benzoyl*: needles from EtOH. M.p. 66°.

*Phenylurethane*: m.p. 102–5°.

*Allophanate*: m.p. 176–5° decomp.

Carothers, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1681.

Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 905.

Tiffeneau, Führer, *Bull. soc. chim.*, 1914, **15**, 172.

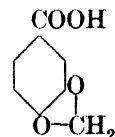
Barger, *J. Chem. Soc.*, 1908, **93**, 567.

Fittig, Remsen, *Ann.*, 1871, **159**, 130, 138.

Decker, Koch, *Ber.*, 1905, **38**, 1741.

Vavon, *Compt. rend.*, 1912, **154**, 361.

**Piperonylic Acid** (3 : 4-Methylenedioxybenzoic acid)



$\text{C}_8\text{H}_6\text{O}_4$  MW, 166

Needles from EtOH, cryst. from  $\text{H}_2\text{O}$ . M.p. 229°. Spar. sol. cold EtOH,  $\text{Et}_2\text{O}$ . Insol. cold  $\text{H}_2\text{O}$ ,  $\text{CHCl}_3$ . Sublimes in prisms.

*Me ester*:  $\text{C}_9\text{H}_8\text{O}_4$ . MW, 180. Needles or leaflets from pet. ether. M.p. 53°. B.p. 273–4° (part. decomp.). Sol. MeOH, EtOH,  $\text{Et}_2\text{O}$ . Volatile in steam.

*Et ester*:  $\text{C}_{10}\text{H}_{10}\text{O}_4$ . MW, 194. Prisms. M.p. 18–5°. B.p. 285°. Sol. EtOH,  $\text{Et}_2\text{O}$ , pet. ether. Insol.  $\text{H}_2\text{O}$ .

*Amide*:  $\text{C}_8\text{H}_7\text{O}_3\text{N}$ . MW, 165. Prisms or

needles from  $\text{H}_2\text{O}$ . M.p.  $169^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ . Insol. pet. ether.

*Chloride*:  $\text{C}_8\text{H}_5\text{O}_3\text{Cl}$ . MW, 184.5. Cryst. M.p.  $80^\circ$ . B.p.  $155^\circ/25$  mm.,  $149-50^\circ/12$  mm.

*Nitrile*:  $\text{C}_8\text{H}_5\text{O}_2\text{N}$ . MW, 147. Needles from  $\text{H}_2\text{O}$ . M.p.  $95^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

Shriner, Kleiderer, *Organic Syntheses*, 1930, X, 82.

Barger, *J. Chem. Soc.*, 1908, 93, 567.

Linge, *Rec. trav. chim.*, 1897, 16, 47.

Rupe, Majewski, *Ber.*, 1900, 33, 3403.

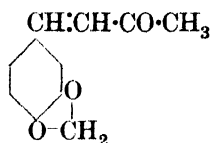
### Piperonylidene-acetaldehyde.

See 3: 4-Methylenedioxybenzaldehyde.

### Piperonylidene-acetic Acid.

See 3: 4-Methylenedioxybenzoic Acid.

**Piperonylideneacetone** (*Methyl 3:4-methylenedioxy-1-phenyl ketone*, 3:4-methylenedioxy-1- $\gamma$ -keto- $\alpha$ -butenylbenzene)



$\text{C}_{11}\text{H}_{10}\text{O}_3$

MW, 190

*Cis*:

Yellow cryst. M.p.  $110-11^\circ$ .

*Trans*:

Colourless cryst. M.p.  $110-11^\circ$ .

Sol. most org. solvents. Spar. sol. boiling  $\text{H}_2\text{O}$ . Volatile in steam.

*Oxime*: white cryst. from EtOH. M.p. about  $186^\circ$ .

*Semicarbazone*: two forms. (i) Given by *cis*- and *trans*-forms. Colourless cryst. from  $\text{CHCl}_3$ . M.p.  $217^\circ$ . Ultra-violet light  $\rightarrow$  (ii). (ii) Yellow cryst. from  $\text{C}_6\text{H}_6$ -pet. ether. M.p.  $168^\circ$ .

*Phenylhydrazone*: prisms. M.p. about  $166^\circ$ .

*Note*.—The configuration of the above three derivatives is not known.

McGookin, Heilbron, *J. Chem. Soc.*, 1924, 125, 2101.

Vavon, Faillebin, *Compt. rend.*, 1919, 169, 67.

Wilson, Heilbron, Sutherland, *J. Chem. Soc.*, 1914, 105, 2895.

Haber, *Ber.*, 1891, 24, 620.

Nomura, Mozawa, *Chem. Abstracts*, 1919, 13, 118.

### $\omega$ -Piperonylideneacetophenone.

See 3: 4-Methylenedioxychalcone.

### Piperonylideneaniline.

See under Piperonal.

### Piperonyloin.

See Piperoin.

### Piperylene.

See 1: 3-Pentadiene.

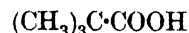
### Piperylpiperidine.

See Piperine.

### Pipitzahoic Acid.

See Perezone.

### Pivalic Acid (*Trimethylacetic acid*)



$\text{C}_5\text{H}_{10}\text{O}_2$

MW, 102

M.p.  $35.3-35.5^\circ$ . B.p.  $163.7-163.8^\circ$ ,  $75-8^\circ/20$  mm.  $D^{20}_4$  0.905.

*Mercuric salt*: needles from  $\text{CHCl}_3$ . M.p.  $235^\circ$ .

*Me ester*:  $\text{C}_6\text{H}_{12}\text{O}_2$ . MW, 116. B.p.  $100-2^\circ$ .  $D^{20}_4$  0.891.

*Et ester*:  $\text{C}_7\text{H}_{14}\text{O}_2$ . MW, 130. B.p.  $118.5^\circ$ .  $D^{20}_4$  0.875.

*p*-Bromophenacyl ester: m.p.  $76.5^\circ$ .

*Anhydride*:  $\text{C}_{10}\text{H}_{18}\text{O}_3$ . MW, 186. B.p.  $190^\circ$ .

*Chloride*:  $\text{C}_5\text{H}_9\text{OCl}$ . MW, 120.5. B.p.  $105-6^\circ$ .

*Amide*:  $\text{C}_5\text{H}_{11}\text{ON}$ . MW, 101. Needles or plates. M.p.  $153-4^\circ$ . B.p. about  $212^\circ$ .

*Nitrile*:  $\text{C}_5\text{H}_9\text{N}$ . MW, 83. Cryst. M.p.  $15-16^\circ$ . B.p.  $105-6^\circ$ .

*Anilide*: m.p.  $127-9^\circ$ .

Hardy, *J. Chem. Soc.*, 1936, 364-5.

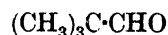
du Pont, U.S.P., 1,995,930, (*Chem. Zentr.*, 1935, II, 594).

Sandborn, Bousquet, *Organic Syntheses*, 1928, VIII, 108.

Puntambeker, Zoellner, *ibid.*, 104.

Butlerow, *Ann.*, 1874, 173, 355; 1873, 170, 158.

### Pivalic Aldehyde (*Trimethylacetaldehyde*)



$\text{C}_5\text{H}_{10}\text{O}$

MW, 86

Constituent of wood spirit. Liq. Solidifies on cooling. M.p.  $6^\circ$ . B.p.  $74-6^\circ$ .  $D^{17}_4$  0.7923. Oxidises in air. Decomp. in daylight.

*Trimer*:  $\text{C}_{15}\text{H}_{30}\text{O}_3$ . MW, 258. Needles from EtOH. M.p.  $82^\circ$ .

*Di-Et acetal*:  $\text{C}_9\text{H}_{20}\text{O}_2$ . MW, 160. Liq. B.p.  $146-8^\circ/742$  mm.  $D^{25}_4$  0.8192.  $n_D$  1.3942.

*Oxime*: m.p.  $41^\circ$ . B.p.  $65^\circ/20$  mm.

*Semicarbazone*: m.p.  $191^\circ$ .

*Anil*: b.p.  $101-2^\circ/20$  mm.

Azine : m.p. 79°.

Dunbar, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 444.

Conant, Webb, Mendum, *J. Am. Chem. Soc.*, 1929, **51**, 1250.

**Pivalone** (*Hexamethylacetone, di-tert.-butyl ketone, 2 : 2 : 4 : 4-tetramethylpentanone-3*)

$(\text{CH}_3)_3\text{C}\cdot\text{CO}\cdot\text{C}(\text{CH}_3)_3$  MW, 142  
 $\text{C}_9\text{H}_{18}\text{O}$

Liq. B.p. 152°, 70°/43 mm.  $D_{20}^{25}$  0.824.  $n_D^{25}$  1.4195. Does not react with usual ketonic reagents.

Vavon, Ivanoff, *Compt. rend.*, 1923, **177**, 453.

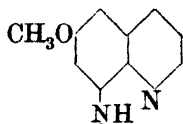
Haller, Bauer, *Compt. rend.*, 1910, **150**, 584.

Henderson, Henderson, Heilbron, *Ber.*, 1914, **47**, 887.

### Pivalophenone.

See *tert.-Butyl phenyl Ketone*.

**Plasmoquin** (*Plasmochin, N-(4-diethylamino-1-methylbutyl)-6-methoxy-8-aminoquinoline*)



$\text{H}_3\text{C}\cdot\text{CH}\cdot[\text{CH}_2]_3\cdot\text{N}(\text{C}_2\text{H}_5)_2$  MW, 315  
 $\text{C}_{19}\text{H}_{29}\text{ON}_3$

Used as an antimalarial.

Knunyantz, Topchiev, Chelintzev, *Chem. Abstracts*, 1934, **28**, 4837.

### Platyphylline

$\text{C}_{18}\text{H}_{25}\text{O}_5\text{N}$  MW, 335

Alkaloid from *Senecio platyphyllus* D.C. Needles from  $\text{H}_2\text{O}$ . M.p. 124–5°. Very sol.  $\text{CHCl}_3$ . Sol. EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. hot  $\text{H}_2\text{O}$ , pet. ether.  $[\alpha]_D - 45.09^\circ$ .

*Perchlorate*: prisms from  $\text{H}_2\text{O}$ . M.p. 222–3° decomp.

*B.HAuCl<sub>4</sub>*: yellow needles +  $\frac{1}{2}\text{H}_2\text{O}$  from EtOH.Aq. M.p. 200–1° decomp.

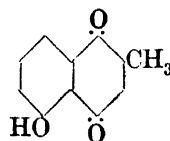
*Methiodide*: needles from EtOH. M.p. 216–17°.  $[\alpha]_D - 31.27^\circ$ .

*Picrolonate*: dark yellow needles from EtOH. M.p. 205–6° decomp.

Orechoff, Konowalowa, Tiedebel, *Ber.*, 1935, **68**, 1886.

Orechoff, *ibid.*, 653.

**Plumbagin** (*5-Hydroxy-2-methyl-1 : 4-naphthoquinone*)



$\text{C}_{11}\text{H}_8\text{O}_3$  MW, 188

Constituent of various species of *Plumbago*. Yellow needles from EtOH.Aq. M.p. 78–9°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ , AcOH,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol. cold  $\text{H}_2\text{O}$ . Volatile in steam. Sublimes.

*Dioxime*: yellow needles from EtOH. M.p. 220°.

*Semicarbazone*: light brown needles from Py-EtOH. M.p. above 280°.

*Phenylhydrazone*: reddish-brown needles from EtOH. M.p. 198°.

*Acetyl*: yellow needles. M.p. 117–18° (138°).

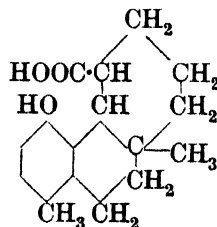
*Benzoyl*: yellow prisms from EtOH.Aq. M.p. 147°.

Fieser, Dunn, *J. Am. Chem. Soc.*, 1936, **58**, 572.

Buruaga, Verdú, *Chem. Zentr.*, 1935, I, 3146.

Roy, Dutt, *J. Indian Chem. Soc.*, 1928, **5**, 419.

**Podocarpic Acid** (1 : 2 : 3 : 4 : 4a : 9 : 10 : 10a-Octahydro - 5 - hydroxy - 8 : 10a - dimethylphenanthrene-4-carboxylic acid)



Probable structure

$\text{C}_{17}\text{H}_{22}\text{O}_3$  MW, 274

Occurs in *Podocarpus cupressina*, var. *imbricata*. Plates from EtOH.Aq. M.p. 193.5°. Sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH. Insol.  $\text{H}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ .  $[\alpha]_{5461} + 165^\circ$  in EtOH.

*Me ester*:  $\text{C}_{18}\text{H}_{24}\text{O}_3$ . MW, 288. Cryst. from EtOH. M.p. 208°. *Me ether*: m.p. 128°.

*Benzoyl*: m.p. 143°.

*Et ester*:  $\text{C}_{19}\text{H}_{26}\text{O}_2$ . MW, 302. Needles. M.p. 161°.

*p-Nitrobenzyl ester*: m.p. 204°.

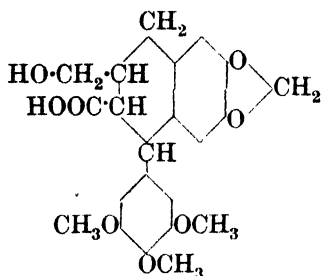
*Me ether*:  $\text{C}_{18}\text{H}_{24}\text{O}_3$ . MW, 288. M.p. 157–8°.

*Acetyl*: sinters at 100°. M.p. 152°.

Sherwood, Short, *Chem. Abstracts*, 1934, **28**, 6435.

Oudemans, *Ann.*, 1873, **170**, 214.

### Podophyllic Acid



$C_{22}H_{24}O_9$

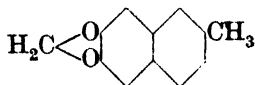
MW, 432

Needles from  $CHCl_3$  or  $EtOH-C_6H_6$ . M.p. 163–5°. Sol.  $EtOH$ . Spar. sol.  $H_2O$ ,  $Et_2O$ ,  $AcOEt$ ,  $CHCl_3$ ,  $CCl_4$ ,  $C_6H_6$ .  $[\alpha]_D^{25} -102.8^\circ$  in  $EtOH$ .

*Hydrazide*: plates from  $H_2O$ . M.p. 155–60°.

Borsche, Niemann, *Ann.*, 1932, **494**, 136.  
Mellanoff, Schaeffer, *Chem. Zentr.*, 1927, II, 1589.

### Podophyllomerol (6 : 7-Methylenedioxy-2-methylnaphthalene)



$C_{12}H_{10}O_2$

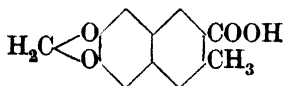
MW, 186

Plates from  $MeOH$ . M.p. 129–129.5°.

*Picrate*: orange-red needles. M.p. 133–4°.

Borsche, Niemann, *Ann.*, 1932, **499**, 67.

### Podophyllomeronic Acid (Methylene ether of phyllomeronic acid, 6 : 7-methylenedioxy-3-methyl-2-naphthoic acid)



$C_{18}H_{10}O_4$

MW, 230

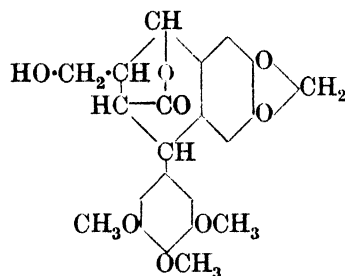
Plates from  $AcOH$ . M.p. 239–40°.

*Me ester*:  $C_{14}H_{12}O_4$ . MW, 244. Needles from  $MeOH$ . M.p. 125–6°.

Borsche, Niemann, *Ann.*, 1932, **494**, 141; 1932, **499**, 67.

Späth, Wessely, Kornfeld, *Ber.*, 1932, **65**, 1547.

### Podophyllotoxin



$C_{22}H_{22}O_8$

MW, 414

Occurs in *Podophyllum*. Needles from  $Me_2CO.Aq.$  or  $MeOH$ . M.p. 114–18°. (M.p. anhyd. 157°.) Sol.  $EtOH$ ,  $Me_2CO$ ,  $AcOH$ ,  $CHCl_3$ , hot  $C_6H_6$ . Spar. sol.  $EtOH.Aq.$  Insol.  $H_2O$ ,  $Et_2O$ , ligroin,  $C_6H_6$ .  $[\alpha]_D^{25} -101.3^\circ$  in  $EtOH$ .

*Acetyl*: needles from  $MeOH$ . M.p. 204°.  $[\alpha]_D^{25} -134.9^\circ$  in  $CHCl_3$ .

Späth, Wessely, Kornfeld, *Ber.*, 1932, **65**, 1542.

Borsche, Niemann, *Ann.*, 1932, **494**, 131; 1932, **499**, 63.

Mellanoff, Schaeffer, *Chem. Zentr.*, 1927, II, 1589.

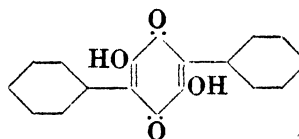
### $\mu$ -Polychloroprene.

See under Chloroprene.

### Polyoxymethylene.

See under Formaldehyde.

### Polyporic Acid (3 : 6-Dihydroxy-2 : 5-diphenyl-p-benzoquinone)



$C_{18}H_{12}O_4$

MW, 292

Occurs in *Polyporus*. Bronze plates from toluene. M.p. 305°. Spar. sol.  $EtOH$ ,  $CHCl_3$ . Insol.  $H_2O$ ,  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ . Sublimes.

*Di-Me ether*:  $C_{20}H_{16}O_4$ . MW, 320. Red cryst. from  $EtOH$ . M.p. 187°.

*Di-Et ether*:  $C_{22}H_{20}O_4$ . MW, 348. Yellow needles or orange-red prisms from  $EtOH$ . M.p. 134°.

*Diacetyl*: needles from *n*-butyl alcohol. M.p. 246° (209°).

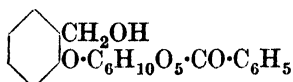
Shildneck, Adams, *J. Am. Chem. Soc.*, 1931, **53**, 2377.

Kögl, *Ann.*, 1926, **447**, 78.

Stahlschmidt, *Ann.*, 1877, **187**, 177.

**Polystichocitrin.**

See Flavaspidic Acid.

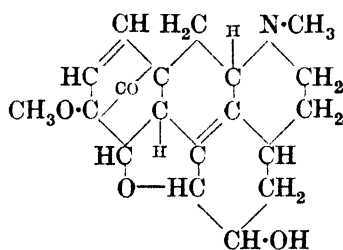
**Populin (6-Benzoylsalicin)** $C_{20}H_{22}O_8$ 

MW, 390

Occurs in leaves and bark of *Populus tremula* and in buds of *P. nigra*, *P. pyramidalis*, etc. Needles +  $2H_2O$  from  $H_2O$ , prisms from EtOH. M.p.  $180^\circ$ . Sol. hot  $H_2O$ , EtOH. Spar. sol. cold  $H_2O$ .  $[\alpha]_D -53^\circ$ ,  $-2.0^\circ$  in Py. Conc.  $H_2SO_4 \rightarrow$  red col.  $Ba(OH)_2 \rightarrow$  salicin.

*Tetra-Me ether*:  $C_{24}H_{30}O_8$ . MW, 446. Needles from Et<sub>2</sub>O-ligroin. M.p.  $134-5^\circ$ .  $[\alpha]_D -31.7^\circ$  in  $CHCl_3$ .

Richtinger, Yeakel, *J. Am. Chem. Soc.*, 1934, **56**, 2495.

**Porphyroxine** $C_{19}H_{23}O_4N$ 

MW, 329

Prisms from ligroin. M.p.  $134-5^\circ$ . Sol.  $CHCl_3$ ,  $Me_2CO$ , AcOH,  $CS_2$ . Mod. sol. MeOH, AcOEt,  $CCl_4$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ .  $[\alpha]^{32} -139.9^\circ$  in  $CHCl_3$ .

*B, HCl*: needles from  $H_2O$ . M.p.  $155^\circ$ .  $[\alpha]^{32} -118.8^\circ$  in  $H_2O$ .

*B, HBr*: needles. M.p.  $148-50^\circ$ .  $[\alpha]^{32} -90.6^\circ$  in  $H_2O$ .

*B, HI*: decomp. at  $115^\circ$ .  $[\alpha]^{32} -77.8^\circ$  in  $H_2O$ .

*B<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>*: plates from  $H_2O$ . M.p.  $193^\circ$ .

*Methiodide*: m.p.  $150-2^\circ$ .

*Methosulphate*: needles from MeOH. M.p.  $205^\circ$  decomp.

*Oxime*: m.p.  $198^\circ$  decomp.

*Phenylhydrazone*: m.p.  $150^\circ$  decomp.

*Semicarbazone*: decomp. at  $244^\circ$ .

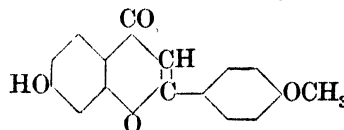
*Acetyl*: m.p.  $125^\circ$ . *B, HCl*: needles. M.p.  $126^\circ$  decomp. *B, HBr*: m.p.  $155^\circ$  decomp.

*B<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>*: m.p.  $190^\circ$  decomp.

*Me ether*:  $C_{20}H_{25}O_4N$ . MW, 343. Cryst. from pet. ether. M.p.  $125-6^\circ$ . *Oxime*: needles from MeOH. M.p.  $185-6^\circ$ . *Phenylhydrazone*:

m.p.  $189^\circ$  decomp. *Semicarbazone*: cryst. from EtOH. M.p.  $217^\circ$ .

Rakshit, *J. Chem. Soc.*, 1919, **115**, 455; *Ber.*, 1926, **59**, 2473.

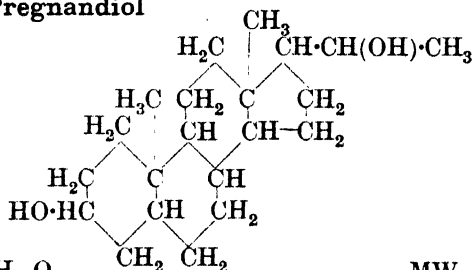
**Pratol (7-Hydroxy-4'-methoxyflavone)** $C_{16}H_{12}O_4$ 

MW, 268

Occurs in flowers of *Trifolium* species. Pale yellow needles from EtOH. M.p.  $263-4^\circ$ . Sol. hot EtOH. Spar. sol.  $H_2O$ , Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ . *Acetyl*: needles or hexagonal plates from EtOH. M.p.  $176-7^\circ$ .

Baker, *J. Chem. Soc.*, 1933, 1386.

Power, Salway, *J. Chem. Soc.*, 1910, **97**, 233, 1008.

**Pregnandiol** $C_{21}H_{36}O_2$ 

MW, 320

Isolated from urine of pregnancy. Plates from EtOH or  $Me_2CO$ . M.p.  $234-5^\circ$ . Spar. sol. most org. solvents. Gives none of recognised sterol colour reactions.

*20-Acetyl*: cryst. from EtOH or  $Me_2CO$ . Aq. M.p.  $170-5^\circ$ .

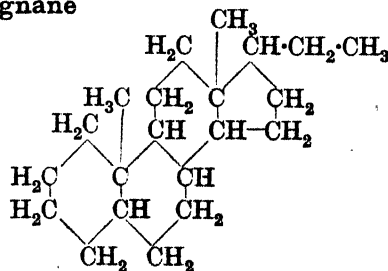
*Diacetyl*: needles from EtOH. M.p.  $180^\circ$ .

Butenandt, Schmidt, *Ber.*, 1934, **67**, 1895.

Butenandt, Hildebrandt, Brucher, *Ber.*, 1931, **64**, 2529.

Butenandt, *Ber.*, 1930, **63**, 661.

Marrian, *Biochem. J.*, 1929, **23**, 1090.

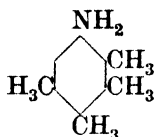
**Pregnane** $C_{21}H_{36}$ 

MW, 288

Cryst. from MeOH. M.p.  $83.5^{\circ}$ .  $[\alpha]_D^{20} + 21.2^{\circ}$  in  $\text{CHCl}_3$ .

Butenandt, Hildebrandt, Brücher, *Ber.*, 1931, **64**, 2538.

**Prehnidine** (2 : 3 : 4 : 5-Tetramethylaniline, 5-amino-1 : 2 : 3 : 4-tetramethylbenzene, 5-amino-prehnitene)



$\text{C}_{10}\text{H}_{15}\text{N}$  MW, 149

Leaflets from  $\text{H}_2\text{O}$ . M.p.  $70^{\circ}$  ( $64-6^{\circ}$ ). B.p.  $259-60^{\circ}$ . Sol. EtOH,  $\text{Et}_2\text{O}$ , pet. ether. Mod. sol. hot  $\text{H}_2\text{O}$ .

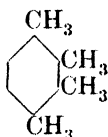
N-Formyl: needles from  $\text{H}_2\text{O}$ . M.p.  $143-4^{\circ}$ .

N-Acetyl: needles from EtOH.Aq. M.p.  $172^{\circ}$  ( $169.5^{\circ}$ ).

Limpach, *Ber.*, 1888, **21**, 644.

Töhl, *ibid.*, 905.

**Prehnitene** (Prehnitol, 1 : 2 : 3 : 4-tetramethylbenzene)



$\text{C}_{10}\text{H}_{14}$  MW, 134

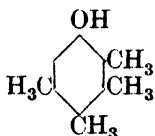
Liq. Solidifies on cooling. M.p.  $-6.4^{\circ}$ . B.p.  $203-4^{\circ}$ ,  $96.4^{\circ}/25$  mm.,  $75-75.5^{\circ}/6.5$  mm.  $D_4^{20}$  0.901.  $n_D^{25}$  1.5196.

Picrate: yellow prisms from EtOH. M.p.  $92-5^{\circ}$ .

Smith, Cass, *J. Am. Chem. Soc.*, 1932, **54**, 1620.

Baril, Hauber, *J. Am. Chem. Soc.*, 1931, **53**, 1089.

**Prehnitenol** (2 : 3 : 4 : 5-Tetramethylphenol, 5-hydroxy-prehnitene, 5-hydroxy-1 : 2 : 3 : 4-tetramethylbenzene)



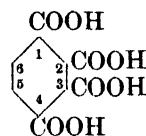
$\text{C}_{10}\text{H}_{14}\text{O}$  MW, 150

Needles from EtOH.Aq. or ligroin. M.p.  $86-7^{\circ}$ . B.p.  $266^{\circ}$ . Very sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin. Volatile in steam.

Acetyl: prisms from ligroin. M.p.  $56-7^{\circ}$ .

Bamberger, Blangey, *Ann.*, 1911, **384**, 307.  
Töhl, *Ber.*, 1888, **21**, 907.

**Prehnitic Acid** (Benzene-1 : 2 : 3 : 4-tetracarboxylic acid)



$\text{C}_{10}\text{H}_6\text{O}_8$  MW, 254

Prisms from  $\text{H}_2\text{O}$ . M.p.  $238^{\circ}$ .

1 : 4-Di-Me ester:  $\text{C}_{12}\text{H}_{10}\text{O}_8$ . MW, 282. M.p.  $176-7^{\circ}$ .

Tetra-Me ester:  $\text{C}_{14}\text{H}_{14}\text{O}_8$ . MW, 310. M.p.  $133-5^{\circ}$ .

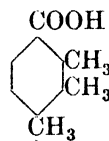
Smith, Byrkil, *J. Am. Chem. Soc.*, 1933, **55**, 4305.

Doebner, *Ann.*, 1900, **311**, 143.

Baeyer, *Ann.*, 1873, **166**, 325.

Meyer, Sudborough, *Ber.*, 1894, **27**, 1591.

**Prehnitylic Acid** (2 : 3 : 4-Trimethylbenzoic acid)



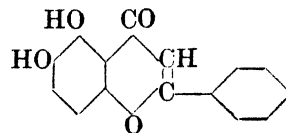
$\text{C}_{10}\text{H}_{12}\text{O}_2$  MW, 164

Prisms from EtOH. M.p.  $167.5^{\circ}$ .  $k = 3.49 \times 10^{-5}$  at  $25^{\circ}$ . Mod. volatile in steam.

Jacobsen, *Ber.*, 1886, **19**, 1214.

Lapworth, Chapman, *J. Chem. Soc.*, 1900, **77**, 316.

**Primetin** (5 : 6-Dihydroxyflavone)



$\text{C}_{15}\text{H}_{10}\text{O}_4$  MW, 254

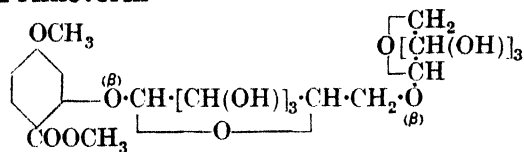
Obtained from *Primula modesta*. Yellow prisms from EtOH. M.p.  $230-1^{\circ}$ . Alc.  $\text{FeCl}_3 \rightarrow$  green col. Sol. alkalis  $\rightarrow$  red sols. Sol. conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.

Mono-Me ether:  $\text{C}_{16}\text{H}_{12}\text{O}_4$ . MW, 268. Needles from EtOH. M.p.  $211-12^{\circ}$ . Insol. alkalis.  $\text{FeCl}_3 \rightarrow$  brownish-violet col. Acetyl: needles from EtOH. M.p.  $176-7^{\circ}$ .

Diacetyl: needles from EtOH. M.p.  $189^{\circ}$ .

Nagai, Hatori, *Chem. Zentr.*, 1930, **II**, 409.

## Primeverin

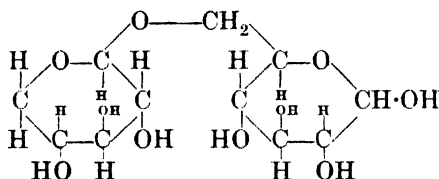
 $C_{20}H_{28}O_{13}$ 

MW, 476

A glucoside from the roots of *Primula officinalis*. Needles from MeOH or EtOH. M.p. 205°.  $[\alpha]_D^{20} - 76.75^\circ$  in  $H_2O$ . Hyd.  $\rightarrow$  4-methoxysalicylic acid + xylose + glucose.

Hexa-acetyl: needles from dil. MeOH or dil. EtOH. M.p. 125°.

Jones, Robertson, *J. Chem. Soc.*, 1933, 1618.

Primeverose (Glucose-6- $\beta$ -d-xyloside) $C_{11}H_{20}O_{10}$ 

MW, 312

Obtained from various glucosides e.g. primeverin and primulaverin from *Primula officinalis*, genticaulin from *Gentiana lutea*, rhamnucosin from *Rhamnus catharticus*. Cryst. M.p. 210° (208°). Sweet taste. Sol.  $H_2O$ . Spar. sol. EtOH.  $[\alpha]_D^{20} + 23.8^\circ \rightarrow -3.4^\circ$  in  $H_2O$ . Reduces Fehling's. Dil. acids  $\rightarrow$  xylose + d-glucose. Br water  $\rightarrow$  primeverobionic acid.

Phenylosazone: yellow needles from  $H_2O$ . M.p. 220°. Sol. EtOH,  $Me_2CO$ . Insol.  $Et_2O$ ,  $CHCl_3$ .  $[\alpha]_D^{19} - 109.7^\circ$  in Py.

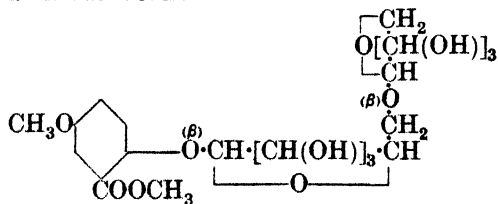
$\beta$ -Hepta-acetyl: needles from EtOH. M.p. 216°. Sol.  $CHCl_3$ . Spar. sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .  $[\alpha]_D^{20} - 23.5^\circ$  in  $CHCl_3$ .

Helferich, Rauch, *Ann.*, 1927, 455, 168.

Goris, Vischniac, *Compt. rend.*, 1919, 169, 871, 975.

Bridel, *Compt. rend.*, 1925, 180, 1421.

## Primulaverin

 $C_{20}H_{28}O_{13}$ 

MW, 476

M.p. 163°.  $[\alpha]_D - 66.51^\circ$ . Hyd.  $\rightarrow$  5-methoxysalicylic acid + xylose + glucose.

Goris, Vischniac, *Bull. soc. chim.*, 1920, 27, 262.

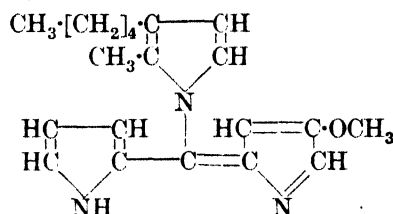
## Primulin chloride

See Cinin chloride.

## Procaine.

See Novocaine.

## Prodigiosine

 $C_{20}H_{25}ON_3$ 

MW, 323

Red pigment of *Bacillus prodigiosus*. Brittle solid. Sinters at 70-80°. No definite m.p. Sol. most org. solvents. Insol.  $H_2O$ .

$B.HClO_4$ : needles with metallic lustre from EtOH. Sinters at 226°. M.p. 228°.

Monobenzoyl deriv.: needles from EtOH.Aq. M.p. 170°.

Monosalicyloyl deriv.: needles from EtOH.Aq. M.p. 178°.

Picrate: needles from EtOH. M.p. 176°.

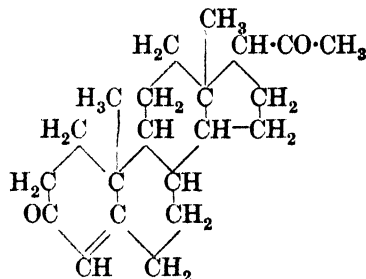
Wrede, Rothhaas, *Z. physiol. Chem.*, 1934, 226, 95.

Wrede, Hettche, *Ber.*, 1929, 62, 2678.

## Proflavine.

See under 2: 8-Diaminoacridine.

## Progesterone (Progestine)

 $C_{21}H_{30}O_2$ 

MW, 314

Obtained from *Corpus luteum*. Exists in two forms of the same physiological activity.

( $\alpha$ ) Prisms from EtOH. M.p. 128.5°. ( $\beta$ ) Needles from pet. ether. M.p. 121-2°.

Readily sol. most org. solvents. Spar. sol.  $H_2O$ .  $[\alpha]_D + 192^\circ$ . Absorption maximum at 240 m $\mu$ . Induces proliferation of uterine muco.

*Dioxime*: cryst. from EtOH.Aq. M.p. 243°.

Butenandt, Westphal, Hohlweg, *Z. physiol. Chem.*, 1934, **227**, 84.

Wintersteiner, Allen, *J. Biol. Chem.*, 1934, **107**, 321.

Fieser, *Chemistry of Natural Products Related to Phenanthrene*, p. 188. (Reinhold Publishing Corporation, New York.)

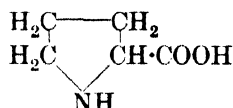
**Progesterone.**

See Progesterone.

**Progynon.**

See Estrone.

**Proline** (*Pyrrolidine-2-carboxylic acid*)



$\text{C}_5\text{H}_9\text{O}_2\text{N}$

MW, 115

*d*l.

Hygroscopic prisms from EtOH-Et<sub>2</sub>O. M.p. 215–20° decomp.  $[\alpha]_D^{20} + 81.9^\circ$  in H<sub>2</sub>O.

*Me ester*:  $\text{C}_6\text{H}_{11}\text{O}_2\text{N}$ . MW, 129.  $[\alpha]_D^{25} + 34^\circ$ .

*N*-p-Toluenesulphonyl: m.p. 130–3°.

*N*-m-Nitrobenzoyl: prisms from H<sub>2</sub>O. M.p. 137–40°.  $[\alpha]_D^{20} + 120^\circ$  in *N*-NaOH.

*l*.

Cryst. from EtOH-Et<sub>2</sub>O. Decomp. at 220–2°.  $[\alpha]_D^{20} - 80.9^\circ$  in H<sub>2</sub>O.

*Amide*:  $\text{C}_5\text{H}_{10}\text{ON}_2$ . MW, 114. *N*-Acetyl: m.p. 178–80°.

*Anhydride*:  $\text{C}_{10}\text{H}_{14}\text{O}_2\text{N}_2$ . MW, 194. M.p. 149°.  $[\alpha]_D^{19} - 147.2^\circ$ .

*N*:2 : 4-Dinitrophenyl: m.p. 136°.

*N*-p-Toluenesulphonyl: m.p. 130–3°.

*Picrate*: m.p. 153–4°.

*dl*.

Hygroscopic needles from EtOH-Et<sub>2</sub>O. M.p. 205° decomp.

*Hydrate*:  $\text{C}_{10}\text{H}_{18}\text{O}_4\text{N}_2$ , H<sub>2</sub>O. M.p. 190–1°. Sol. H<sub>2</sub>O, EtOH. Spar. sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. Et<sub>2</sub>O.

*B.HCl*: cryst. from H<sub>2</sub>O. M.p. 158–9°.

*Et ester*:  $\text{C}_7\text{H}_{13}\text{O}_2\text{N}$ . MW, 143. Oil. B.p. 80°/13 mm.

*Amide*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 93°. Sol. EtOH, AcOEt, CHCl<sub>3</sub>.

*Anhydride*: needles. M.p. 183–4°.

*Anilide*: cryst. from Me<sub>2</sub>CO. M.p. 170°.

*N*-Me: see Hygic Acid.

*N*-m-Nitrobenzoyl: prisms from H<sub>2</sub>O. M.p. 90–2°.

*Picrate*: cryst. from EtOH. M.p. 135–7°.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 1122.

Fischer, Zemplén, *Ber.*, 1909, **42**, 2997.

Kapfhammer, Eck, *Z. physiol. Chem.*, 1927, **170**, 294.

Kapfhammer, Matthes, *Z. physiol. Chem.*, 1933, **223**, 43.

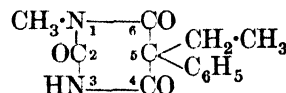
Jacobs, Craig, *J. Biol. Chem.*, 1935, **110**, 521.

Grassmann, Armin, *Ann.*, 1935, **519**, 192.

Putochin, *Ber.*, 1926, **59**, 1987; 1923, **56**, 2213.

Abderhalden, Sickel, *Z. physiol. Chem.*, 1926, **159**, 163.

**Prominal** (1-Methyl-5-ethyl-5-phenylbarbituric acid)



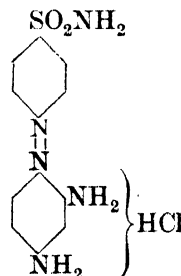
$\text{C}_{13}\text{H}_{14}\text{O}_3\text{N}_2$

MW, 246

M.p. 176°.

I.G., F.P., 753,178, (*Chem. Zentr.*, 1934, I, 895).

**Prontosil** (4'-Sulphamido-2 : 4-diaminoazobenzene hydrochloride, Streptozon, Rubiazol, Pron-tosil Red)



$\text{C}_{12}\text{H}_{13}\text{O}_2\text{N}_5$

MW, 259

Cryst. powder. M.p. 247–51°. Sol. 400 parts H<sub>2</sub>O. One of a series of substances possessing strong bactericidal properties. Used for treatment of streptococcal and staphylococcal infections, especially erysipelas.

*p*-Aminobenzenesulphonamide, possessing similar physiological properties, is sometimes referred to as "Prontosil."

Domagk, *Angew. Chem.*, 1935, **48**, 660.

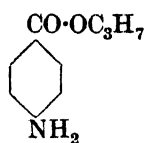
Goissedet, Despois, Gaillot, Mayer, *Compt. Rend. Société Biologique*, 1936, **121**, 1082.

**Propadiene.**

See Allene.



**Propæsin** (*p*-Aminobenzoic acid propyl ester)



$\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$

MW, 179

Prisms. M.p.  $73-4^\circ$ . Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Local anæsthetic.

Fritzsche, D.R.P., 213,459, (*Chem. Zentr.*, 1909, II, 1025).

**Propaldehyde.**

See Propionaldehyde.

**Propanal.**

See Propionaldehyde.

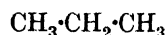
**Propanalone.**

See Pyruvic Aldehyde.

**Propandial-1 : 3.**

See Glutaraldehyde.

**Propane**



$\text{C}_3\text{H}_8$

MW, 44

Occurs in petroleum gas. F.P.  $-189.9^\circ$ . B.p.  $-44.5^\circ$ ,  $-124.2^\circ/3$  mm.  $D_4^{20}$  0.536,  $D_4^{15.9}$  0.515. Heat of comb.  $\text{C}_p$  529.21 Cal. (528.4 Cal.).

Maass, Wright, *J. Am. Chem. Soc.*, 1921, **43**, 1098.

Spencer, Price, *J. Chem. Soc.*, 1910, **97**, 388.

Wolkow, Menschutkin, *Chem. Zentr.*, 1900, II, 42.

Timmermans, *Compt. rend.*, 1914, **158**, 790.

Meyer, *Ber.*, 1893, **26**, 2071.

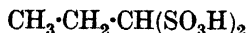
**Propane-dicarboxylic Acid.**

See Ethylmalonic Acid, Methylsuccinic Acid, and Glutaric Acid.

**Propane 2 : 2-diethyl disulphone.**

See Sulphonal.

**Propane-1 : 1-disulphonic Acid** (*Ethylmethionic acid*)



$\text{C}_3\text{H}_8\text{O}_6\text{S}_2$

MW, 204

*Diphenyl ester* :  $\text{C}_{15}\text{H}_{14}\text{O}_6\text{S}_2$ . MW, 354. Oil. B.p.  $200^\circ/0.005$  mm.

*Diamide* :  $\text{C}_3\text{H}_{10}\text{O}_4\text{N}_2\text{S}_2$ . MW, 202. M.p.  $169-70^\circ$ .

*Di-trimethylamide* : cryst. from Et<sub>2</sub>O. M.p.  $133^\circ$ .

*Dianilide* : cryst. from EtOH. M.p.  $151-2^\circ$ .

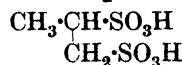
*Di-[N-Et]-anilide* : ethylmethionide. Prisms from EtOH. M.p.  $128-9^\circ$ .

*Di-[N-Et]-phenetidine* : needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p.  $93.5-94.5^\circ$ .

Schroeter, *Ann.*, 1918, **418**, 230.

Klaver, *Rec. trav. chim.*, 1935, **54**, 208.

**Propane-1 : 2-disulphonic Acid**



$\text{C}_3\text{H}_8\text{O}_6\text{S}_2$

MW, 204

Syrup. Sol. H<sub>2</sub>O, EtOH.

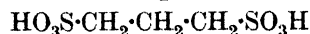
*Dichloride* :  $\text{C}_3\text{H}_6\text{O}_4\text{S}_2\text{Cl}_2$ . MW, 241. Plates from pet. ether. M.p.  $48^\circ$ .

Autenrieth, Rudolph, *Ber.*, 1901, **34**, 3477.

Buckton, Hofmann, *Ann.*, 1856, **100**, 153.

Clutterbuck, Cohen, *J. Chem. Soc.*, 1922, **121**, 120.

**Propane-1 : 3-disulphonic Acid**



$\text{C}_3\text{H}_8\text{O}_6\text{S}_2$

MW, 204

Needles. Decomp. without melting. Sol. H<sub>2</sub>O, EtOH.

*Dichloride* : m.p.  $45^\circ$ .

*Diamide* : cryst. from H<sub>2</sub>O or EtOH. M.p.  $169^\circ$ .

*Dianilide* : m.p.  $129^\circ$ .

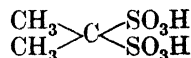
*Dihydrazide* : needles from EtOH. M.p.  $105^\circ$ .

*Di-phenylhydrazide* : needles from Me<sub>2</sub>CO.Aq. M.p.  $177^\circ$  decomp.

See last reference above and also

Autenrieth, Bernheim, *Ber.*, 1904, **37**, 3808.

**Propane-2 : 2-disulphonic Acid** (*Dimethylmethionic acid*)



$\text{C}_3\text{H}_8\text{O}_6\text{S}_2$

MW, 204

*Diphenyl-ester* : m.p.  $96^\circ$ .

*Di-[N-Et]-anilide* : dimethylmethionide. Cryst. from EtOH. M.p.  $130-2^\circ$ .

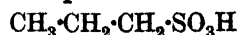
*Di-[N-Me]-phenetidine* : m.p.  $114-15^\circ$ .

*Di-[N-Et]-phenetidine* : m.p.  $109^\circ$ .

Schroeter, *Ann.*, 1919, **418**, 233.

Klaver, *Rec. trav. chim.*, 1935, **54**, 208.

**Propane-1-sulphonic Acid**



$\text{C}_3\text{H}_8\text{O}_3\text{S}$

MW, 124

*Chloride* :  $\text{C}_3\text{H}_7\text{O}_2\text{SCl}$ . MW, 142.5. B.p.  $180^\circ$  decomp.,  $77.3-78^\circ/13$  mm.  $D_4^{15}$  1.2826.

*Amide*:  $C_3H_5O_2NS$ . MW, 123. Prisms from  $Et_2O$ . M.p.  $52^\circ$ . Sol.  $H_2O$ ,  $EtOH$ .

Duguet, *Rec. trav. chim.*, 1902, **21**, 77.

Wagner, Reid, *J. Am. Chem. Soc.*, 1931, **53**, 3411.

### Propane-2-sulphonic Acid



$C_3H_8O_3S$  MW, 124

*Chloride*: oil. B.p.  $79^\circ/18$  mm.

*Amide*: cryst. from  $Et_2O$ -pet. ether. M.p.  $60^\circ$ . Sol.  $EtOH$ ,  $Et_2O$ . Insol.  $C_6H_6$ .

See last reference above and also

Claus, *Ber.*, 1872, **5**, 660; 1875, **8**, 532.

Duguet, *Rec. trav. chim.*, 1906, **25**, 215.

### Propane-1 : 2 : 3-tricarboxylic Acid.

See Tricarballic Acid.

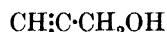
### Propanol.

See Propyl Alcohol and Isopropyl Alcohol.

### Propanolamine.

See 3-Aminopropyl Alcohol.

**Propargyl Alcohol** (3-Hydroxyallylene, hydroxymethylacetylene, acetylenylcarbinol)



$C_3H_4O$  MW, 56

Volatile liq. B.p.  $114-15^\circ$ .  $D_4^{20}$  0.9715.  $n_D^{20}$  1.43064.

*Me ether*: see Methyl propargyl Ether.

*Et ether*:  $C_5H_8O$ . MW, 84. B.p.  $82^\circ$ .  $D_4^{25}$  0.8324.  $n_D^{20}$  1.40390.

*Isoamyl ether*:  $C_8H_{14}O$ . MW, 126. B.p.  $140^\circ$ .  $D_4^{25}$  0.84.

*Phenyl ether*:  $C_9H_8O$ . MW, 132. Liq. Decomp. at  $210^\circ$ .  $D_6^1$  1.246. Insol.  $H_2O$ .

*Acetyl*: b.p.  $124-5^\circ$ .  $D_4^{20}$  1.0052.  $n_D^{20}$  1.42047.

*Phenylurethane*: m.p.  $63^\circ$ .

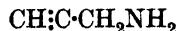
Henry, *Ber.*, 1873, **6**, 729.

Lespieau, *Ann. chim. phys.*, 1912, **27**, 158.

Paal, Heupel, *Ber.*, 1891, **24**, 3039.

Liebermann, Kretschmer, *Ann.*, 1871, **158**, 230.

**Propargylamine** (3-Aminoallylene, 3-amino-propyne)



$C_3H_5N$  MW, 55

*B,HCl*: unstable. Sol.  $H_2O$ ,  $EtOH$ .

*B,HBr*: unstable. M.p.  $171^\circ$ .

*B,HI*: unstable. M.p.  $205^\circ$ .

*B,(COOH)\_2*: m.p.  $143^\circ$ . Sol.  $H_2O$ . Insol.  $EtOH$ .

Dict. of Org. Comp.—III.

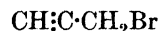
*N-Me*:  $C_4H_7N$ . MW, 69. *B,HI*: m.p.  $83^\circ$ . *Oxalate*: m.p.  $141^\circ$ .

*Picrate*: plates. M.p.  $189^\circ$ .

Paal, Hermann, *Ber.*, 1889, **22**, 3080.

Paal, Heupel, *Ber.*, 1891, **24**, 3040.

**Propargyl bromide** (3-Bromoallylene, 3-bromopropine)



$C_3H_3Br$  MW, 119

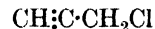
B.p.  $88-90^\circ$  ( $82^\circ$ ),  $35^\circ/130$  mm.  $D_4^{19}$  1.579.  $n_D^{19}$  1.4942.

Henry, *Ber.*, 1874, **7**, 761.

Kinmann, *Bull. soc. chim.*, 1926, **39**, 698.

v. Braun, Kühn, Siddiqui, *Ber.*, 1926, **59**, 1086.

**Propargyl chloride** (3-Chloroallylene, 3-chloropropine)



$C_3H_3Cl$  MW, 74.5

Liq. B.p.  $65^\circ$ .  $D_4^0$  1.0454.

Henry, *Ber.*, 1875, **8**, 398.

### Propargylic Acid.

See Propiolic Acid.

### Propargylic Aldehyde.

See Propiolic Aldehyde.

### Propene.

See Propylene.

### Propenylacetic Acid.

See 2-Ethylidenepropionic Acid.

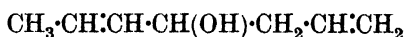
### 2-Propenylacrylic Acid.

See Sorbic Acid.

### 3-Propenylallyl Alcohol.

See 2 : 4-Hexadienol-1.

**Propenylallylcarbinol** (1 : 5-Heptadienol-4, 4-hydroxy-1 : 5-heptadiene)



$C_7H_{12}O$  MW, 112

B.p.  $156-7^\circ$  decomp./735 mm.,  $68-9^\circ/24$  mm.,  $54-5^\circ/6$  mm.  $D_4^{20}$  0.8612.  $n_D^{13.7}$  1.45527. Heat with  $KHSO_4 \rightarrow$  1 : 3 : 5-heptatriene. Oxidises in air.

Auwers, *J. prakt. Chem.*, 1923, **105**, 373.

Enklaar, *Ber.*, 1916, **49**, 211.

I.G., D.R.P., 544,388, (*Chem. Zentr.*, 1932, II, 3156).

### Propenylanisole.

See Anethole and under Propenylphenol.

### Propenylbenzene.

See  $\beta$ -Methylstyrene.

**Propenyl bromide (1-Bromopropylene)**

$\text{C}_3\text{H}_5\text{Br}$  MW, 121  
B.p. 58–60°/747 mm.  $D_4^{20}$  1.4133.  $n_D^{20}$  1.45193. Insol.  $\text{H}_2\text{O}$ . Alc. KOH  $\longrightarrow$  allylene.

Juvala, *Ber.*, 1930, **63**, 1994.

Bachman, *J. Am. Chem. Soc.*, 1933, **55**, 4282.

Schmidt, *Kleine, Ann.*, 1904, **337**, 86.

**1-Propenyl-1-butylene.**

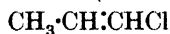
See 2 : 4-Heptadiene.

**Propenylbutyric Acid.**

See 4-Heptenic Acid and 2-Methyl-3-heptenic Acid.

**Propenylcarbinol.**

See Crotonyl Alcohol.

**Propenyl chloride (1-Chloropropylene)**

$\text{C}_3\text{H}_5\text{Cl}$  MW, 76.5

*Cis* :

F.p. – 134.8°. B.p. 32.8°.

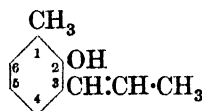
*Trans* :

F.p. – 99°. B.p. 37.4°.

Wislicenus, *Ann.*, 1888, **248**, 297.

Timmermans, *Bull. soc. chim. Belg.*, 1927, **36**, 502.

Reboul, *Ann. chim. phys.*, 1878, **14**, 462.

**3-Propenyl-o-cresol (1-[2-Hydroxy-m-tolyl]-propylene)**

$\text{C}_{10}\text{H}_{12}\text{O}$  MW, 148

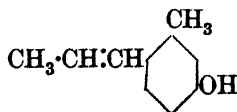
Needles from pet. ether. M.p. 41–2°. B.p. 113–15°/12 mm. Sol. most org. solvents, NaOH.

Auwers, Wittig, *Ber.*, 1924, **57**, 1273.

**5-Propenyl-o-cresol (1-[4-Hydroxy-m-tolyl]-propylene).**

*Me ether* :  $\text{C}_{11}\text{H}_{14}\text{O}$ . MW, 162. B.p. 121–3°/14 mm.  $D_4^{18}$  0.9844.  $n_D^{17}$  1.5570. Nitrosochloride : m.p. 117°.

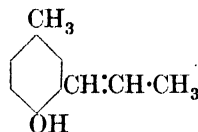
Klages, *Ber.*, 1904, **37**, 3992.

**6-Propenyl-m-cresol (1-[4-Hydroxy-o-tolyl]-propylene)**

$\text{C}_{10}\text{H}_{12}\text{O}$  MW, 148

*Me ether* :  $\text{C}_{11}\text{H}_{14}\text{O}$ . MW, 162. Oil. B.p. 119–21°/13 mm.  $D_4^{18}$  0.9849.  $n_D^{18}$  1.5555. Nitrosochloride : needles. M.p. 108°. Spar. sol.  $\text{Et}_2\text{O}$ .

Klages, *Ber.*, 1904, **37**, 3994.

**3-Propenyl-p-cresol (1-[6-Hydroxy-m-tolyl]-propylene)**

$\text{C}_{10}\text{H}_{12}\text{O}$  MW, 148

B.p. 126–8°/14 mm., 120–4°/11 mm.  $D_4^{20}$  1.019.  $n_D^{20}$  1.5727.

*Me ether* :  $\text{C}_{11}\text{H}_{14}\text{O}$ . MW, 162. B.p. 127.5–128°/19 mm.  $D_4^{20}$  0.979.  $n_D^{20}$  1.5522.

Auwers, *Ann.*, 1917, **413**, 299.

**2-Propenylcrotonic Acid.**

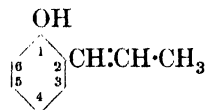
See 2-Methylsorbic Acid.

**Propenylethylidenepropene.**

See 2 : 6-Octadiene.

**Propenylguaiaacol.**

See Isochavibetol and Isoeugenol.

**o-Propenylphenol (o-Hydroxy-β-methylstyrene)**

$\text{C}_9\text{H}_{10}\text{O}$  MW, 134

Needles from ligroin. M.p. 37–8°. B.p. 230–1°, 112–13°/12 mm.  $D_4^{18.9}$  1.0441.  $n_D^{14.2}$  1.584.

*Me ether* : o-propenylanisole.  $\text{C}_{10}\text{H}_{12}\text{O}$ . MW, 148. Volatile oil. B.p. 223–4°/751 mm., 104–5°/13 mm.  $D_4^{18}$  0.9962.  $n_D^{18}$  1.560.

*Et ether* : o-propenylphenetole.  $\text{C}_{11}\text{H}_{14}\text{O}$ . MW, 162. Oil. B.p. 230–1°/757 mm., 120–1°/17 mm.  $D_4^{24}$  0.97307.  $n_D^{24}$  1.544.

Claisen, *Ann.*, 1919, **418**, 86.

Pauly, v. Buttlar, *Ann.*, 1911, **383**, 280.

Klages, *Ber.*, 1904, **37**, 3987.

**m-Propenylphenol (m-Hydroxy-β-methylstyrene).**

*Me ether* : m-propenylanisole. Liq. B.p. 226–9°.  $D_4^0$  1.0013.

*Et ether* : m-propenylphenetole. Volatile oil. B.p. 124–6°/16 mm.  $D_4^{22}$  0.9782.  $n_D^{22}$  1.542. Nitrosochloride : m.p. 122–3°.

Moureu, *Bull. soc. chim.*, 1896, **15**, 1024.

Klages, *Ber.*, 1904, **37**, 3989.

**p-Propenylphenol.**

See Anol.

**Propenylphenylcarbinol** (1-Phenyl-2-but-enol-1,  $\alpha$ -propenylbenzyl alcohol, 1-phenylcrotonyl alcohol) $\text{C}_{10}\text{H}_{12}\text{O}$ 

MW, 148

B.p. 121.5–123.5°/14 mm., 88–90°/1 mm.  
 $n_D^{18}$  1.5412.

Succinate : m.p. 72–80°.

Acid succinate : m.p. 76–8°.

Ingold, Wilson, *J. Chem. Soc.*, 1933, 1497.Burton, *J. Chem. Soc.*, 1929, 457.**2-Propenylpiperidine.**See  $\beta$ -Coniceine.**2-Propenylpropylene.**

See 2-Methyl-1 : 3-pentadiene.

**2-Propenylpyridine** $\text{C}_8\text{H}_9\text{N}$ 

MW, 119

Liq. B.p. 189–90°.  $D_4^{20}$  0.9595. Spar. sol.  $\text{H}_2\text{O}$ . Volatile in steam. $B, H\text{AuCl}_4$  : needles from  $\text{HCl.Aq.}$  M.p. 135–6°. $B_2, H_2\text{PtCl}_6$  : needles. M.p. 185–6° decomp.Ladenburg, *Ann.*, 1888, 247, 26.Note.—This compound is referred to in the above reference as  $\alpha$ -allylpyridine.**4-Propenylpyridine.**

Liq. B.p. 200–2°. Volatile in steam.

 $B, H\text{Cl}$  : cryst. M.p. 247°. $B, H\text{AuCl}_4$  : yellow needles from  $\text{HCl.Aq.}$  M.p. 174° decomp. $B, H\text{Cl}, H\text{gCl}_2$  : needles from  $\text{H}_2\text{O}$ . M.p. 150°. $B_2, H_2\text{PtCl}_6$  : needles. M.p. 206° decomp.Picrate : yellow needles from  $\text{H}_2\text{O}$ . M.p. 169–70° decomp.Ahrens, *Ber.*, 1905, 38, 157.**4-Propenylveratrol.**

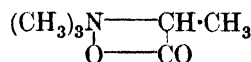
See under Isoeugenol.

**Propine.**

See Allylene.

**Propine-1 : 3-dicarboxylic Acid.**

See Glutinic Acid.

 **$\alpha$ -Propiobetaine** (*Homobetaine*, *alanine-betaine*, *methylbetaine*) $\text{C}_6\text{H}_{13}\text{O}_2\text{N}$ 

MW, 131

l.

Plates from  $\text{EtOH-Et}_2\text{O}$ . Decomp. at 242°.  
 $[\alpha]_D^{20} - 19.7^\circ$  in  $\text{H}_2\text{O}$ . $B, H\text{AuCl}_4$  : dimorphous. (i) Yellow plates from dil.  $\text{HCl}$ . M.p. 259° decomp. (ii) Cryst. from  $\text{H}_2\text{O}$ . M.p. 226° decomp.

dl.

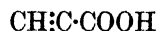
Cryst.

 $B, H\text{AuCl}_4$  : needles from 1%  $\text{HCl}$ . M.p. 240°. $B_2, H_2\text{PtCl}_6$  : orange plates +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. 210–12° decomp.Fischer, *Ber.*, 1907, 40, 5002.Kossel, Edlbacher, *Z. physiol. Chem.*, 1919, 107, 45.**Propiodinitrile** (*Dipropionitrile*, 2-imino-1-methyl-n-valeronitrile) $\text{C}_6\text{H}_{10}\text{N}_2$ 

MW, 110

M.p. 47–8°. B.p. 257–8°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ . At 330°  $\rightarrow$  propionitrile. Na in  $\text{EtOH} \rightarrow$  propylamine. Gradually decomp. by hot  $\text{H}_2\text{O}$ .v. Meyer, *J. prakt. Chem.*, 1888, 38, 338.**Propioin.**

See Diethylketol.

**Propiolic Acid** (*Acetylene-carboxylic acid*, *propargylic acid*) $\text{C}_3\text{H}_2\text{O}_2$ 

MW, 70

Liq. with odour resembling acetic acid. M.p. 18°; monohydrate cryst. felted mass, m.p. 0.3°;  $3\text{C}_3\text{H}_2\text{O}_2 + 1\text{H}_2\text{O}$ , needles, m.p. 10°. B.p. 144° decomp., 102°/200 mm., 83–4°/50 mm. Misc. with  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .  $D_4^{20}$  (anhyd.) 1.1380.  $n_D^{20}$  1.43064. Red.  $\rightarrow$  propionic acid.  $\text{HCl}$ ,  $\text{HBr}$ ,  $\text{HI} \rightarrow \beta$ -halogen-acrylic acid. Reduces  $\text{AgNO}_3$  and  $\text{HgCl}_2$  and platinic chloride sols. in warm. Forms explosive Ag and Cu comps.  $\text{Cu}_2\text{Cl}_2 \rightarrow$  cryst. add. comp. $K$  salt : cryst. +  $1\text{H}_2\text{O}$ . Decomp. at 105°. Very sol.  $\text{H}_2\text{O}$ .

*Aniline salt*: prisms +  $\frac{1}{2}$ H<sub>2</sub>O from EtOH.Aq. M.p. 71–2°.

*Me ester*: C<sub>4</sub>H<sub>4</sub>O<sub>2</sub>. MW, 84. B.p. 102°/742 mm. Lachrymatory.

*Et ester*: C<sub>5</sub>H<sub>6</sub>O<sub>2</sub>. MW, 98. B.p. 119°/745 mm. D<sub>25</sub><sup>25</sup> 0.9583. n<sub>D</sub><sup>15</sup> 1.4133. Red.  $\rightarrow$  ethyl propargyl ether. NH<sub>2</sub>·NH<sub>2</sub>  $\rightarrow$  pyrazolone.

*Anhydride*: C<sub>6</sub>H<sub>2</sub>O<sub>3</sub>. MW, 122. B.p. 56°/16 mm. D<sub>4</sub><sup>21</sup> 1.1432. n<sub>D</sub><sup>23.4</sup> 1.4358. Spar. sol. ligroin.

*Nitrile*: C<sub>3</sub>HN. MW, 51. M.p. 5°. B.p. 42.5°. D<sub>4</sub><sup>17</sup> 0.8159. n<sub>D</sub><sup>17</sup> 1.38699. Spar. sol. H<sub>2</sub>O. Sols. rapidly decompose. Decomp. by air and light. KOH  $\rightarrow$  resin. AgNO<sub>3</sub>  $\rightarrow$  white ppt. Vapour affects mucous membrane.

*Amide*: C<sub>3</sub>H<sub>3</sub>ON. MW, 69. Needles or plates from Et<sub>2</sub>O. M.p. 61–2°. Very sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>, cold C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>.

*Anilide*: needles from H<sub>2</sub>O. M.p. 86–7°.

Backer, Beute, *Rec. trav. chim.*, 1935, 54, 168.

Chéou, *Chem. Abstracts*, 1935, 2513.

Straus, Heyn, Schwener, *Ber.*, 1930, 63, 1090.

Straus, Voss, *Ber.*, 1926, 59, 1685.

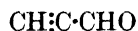
Moureu, André, *Compt. rend.*, 1913, 157, 897.

Moureu, Bongrand, *Compt. rend.*, 1910, 151, 946.

Perkin, Simonsen, *J. Chem. Soc.*, 1907, 91, 834.

Baeyer, *Ber.*, 1885, 18, 677.

### Propiolic Aldehyde (Propargylic aldehyde)



C<sub>3</sub>H<sub>2</sub>O

MW, 54

Thin oil. B.p. 59–61°. Misc. with H<sub>2</sub>O. Affects eyes and mucous membranes. NH<sub>3</sub>·AgNO<sub>3</sub>  $\rightarrow$  white ppt. NH<sub>3</sub>·CuCl<sub>2</sub>  $\rightarrow$  yellowish-red ppt. Dil. alkalis  $\rightarrow$  acetylene + formic acid. NH<sub>2</sub>OH  $\rightarrow$  isoxazole.

*Di-Me acetal*: C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>. MW, 100. B.p. 110°.

*Di-Et acetal*: C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>. MW, 128. B.p. 139–41°. NH<sub>2</sub>OH  $\rightarrow$  isoxazole. NH<sub>2</sub>·NH<sub>2</sub>  $\rightarrow$  pyrazole. *Cu deriv.*: m.p. 160°.

Grard, *Ann. chim.*, 1930, 13, 336.

Claisen, *Ber.*, 1903, 36, 3667; 1898, 31, 1022.

**Propionaldehyde** (Propanal, propaldehyde, propionic aldehyde)



C<sub>3</sub>H<sub>6</sub>O

MW, 58

Liq. with suffocating odour. F.p. – 81°. B.p. 47.5–49°. Sol. 5 parts H<sub>2</sub>O at 20°. D<sub>25</sub><sup>25</sup> 0.79664. n<sub>D</sub><sup>20</sup> 1.3636. Heat of comb. C<sub>p</sub> 434.35 Cal., C<sub>v</sub> 433.8 Cal. HCl  $\rightarrow$  metapropionaldehyde and parapropropionaldehyde. KOH  $\rightarrow$  propionaldol. Red.  $\rightarrow$  propyl alcohol. *m*-Dinitrobenzene + KOH  $\rightarrow$  greenish-brown col. Sodium nitroprusside and piperidine  $\rightarrow$  blue col. (green to blue in dil. sol.). Forms bisulphite comp.

*Metapropionaldehyde*: (C<sub>3</sub>H<sub>6</sub>O)<sub>n</sub>. Cryst. M.p. 180°. Insol. H<sub>2</sub>O. Spar. sol. EtOH. Sublimes. Dist. with little HCl  $\rightarrow$  propionaldehyde.

*Parapropropionaldehyde*: C<sub>9</sub>H<sub>18</sub>O<sub>3</sub>. MW, 174. F.p. – 20° to needles. B.p. 172–3°/773 mm., 85–6°/50 mm. Misc. with EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. D<sub>18</sub><sup>18</sup> 0.9445. Dist. with little HCl or H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  propionaldehyde.

*Propionaldehyde-ammonia*: C<sub>3</sub>H<sub>9</sub>ON. MW, 75. Flocculent ppt. Mod. sol. EtOH, Et<sub>2</sub>O. Spar. sol. ligroin. Gradually decomp. at room temp.

*Bisulphite comp.*: cryst. + 1H<sub>2</sub>O. Spar. sol. EtOH.

*Oxime*: C<sub>3</sub>H<sub>7</sub>ON. MW, 73. M.p. 40°. B.p. 130–2°, 77°/100 mm. D<sub>4</sub><sup>20</sup> 0.9258. n<sub>D</sub><sup>20</sup> 1.4287.

*Semicarbazone*: two forms. (i) Needles from C<sub>6</sub>H<sub>6</sub>–ligroin. M.p. 88–90°. Very easily sol. H<sub>2</sub>O, hot C<sub>6</sub>H<sub>6</sub>. (ii) Plates from H<sub>2</sub>O. M.p. 154°. Mod. sol. C<sub>6</sub>H<sub>6</sub>.

*Phenylhydrazone*: oil. B.p. 205°/108 mm. Becomes red in air. *Picrate*: pale yellow needles from EtOH. M.p. 156–7°.

*Diphenylhydrazone*: needles from EtOH.Aq. M.p. 20–1°.

*Methylphenylhydrazone*: yellow oil. B.p. 198°/170 mm.

*o-Nitrophenylhydrazone*: orange needles from EtOH.Aq. M.p. 72°.

*m-Nitrophenylhydrazone*: orange-yellow plates from EtOH.Aq. M.p. 83°.

*p-Nitrophenylhydrazone*: orange-yellow needles from EtOH.Aq. M.p. 125°.

*o-Tolylsemicarbazone*: plates. M.p. 129–30°.

*p-Tolylsemicarbazone*: plates. M.p. 135–7°.

*Benzoylhydrazone*: prisms from hot H<sub>2</sub>O. M.p. 117°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Easily decomp. by acids and alkalis.

*Cyanhydrin*: see under 1-Hydroxybutyric Acid.

*Di-Me acetal*: C<sub>5</sub>H<sub>12</sub>O<sub>2</sub>. MW, 104. B.p. 89°. D<sub>21</sub><sup>21</sup> 0.849. n<sub>D</sub><sup>21</sup> 1.3799.

*Di-Et acetal*: C<sub>7</sub>H<sub>16</sub>O<sub>2</sub>. MW, 132. B.p. 123°. D<sub>0</sub><sup>0</sup> 0.8825.

*Anil*: propylideneaniline. Dimer.  $C_{16}H_{22}N_2$ . Needles from EtOH. M.p. 103–4°. Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. cold EtOH.

Sah, Shih, *J. Chinese Chem. Soc.*, 1935, **3**, 246.

Bauer, Strauss, *Ber.*, 1932, **65**, 311.

Hurd, Meinert, *Organic Syntheses*, 1932, **XII**, 64.

I.G., B.P., 354,388, (*Chem. Zentr.*, 1931, **II**, 2932).

Kirrmann, *Ann. chim.*, 1929, **11**, 223.

Wood, Comley, *J. Soc. Chem. Ind.*, 1923, **42**, 429t.

Harries, Oppenheim, *Chem. Zentr.*, 1916, **II**, 992.

Nef, *Ann.*, 1904, **335**, 202.

### Propionamide



$C_3H_7ON$

MW, 73

Plates from  $C_6H_6$ . M.p. 81.3°. B.p. 213°. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ . Volatile in steam. Heat of comb.  $C_p$  439.8 Cal. Combines with min. acids, Na and K. Na  $\rightarrow$  sodium dipropionamide. Red.  $\rightarrow$  propylamine and dipropylamine.

*Hg comp.*: plates. M.p. 202°. Spar. sol. cold  $H_2O$ .

*N-Formyl*: needles from  $C_6H_6$ . M.p. 65°. Sol.  $H_2O$ , EtOH,  $Et_2O$ .

*N-Acetyl*: see *N-Acetylpropionamide*.

*N-Benzoyl*: needles or prisms. M.p. 98°.

*N-Di-Me*:  $C_5H_{11}ON$ . MW, 101. B.p. 165–78°.

*N-Di-Et*:  $C_7H_{15}ON$ . MW, 129. B.p. 191°. Sol.  $H_2O$ , acids.

*N-Chloro*: cryst. from  $C_6H_6$ . M.p. 34°.

*N-Bromo*: needles. M.p. 80°. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ . Spar. sol. cold  $C_6H_6$ , pet. ether.

*N-Iodo*: m.p. 128° decomp. Sol. EtOH,  $Me_2CO$ . Mod. sol. AcOH. Spar. sol.  $C_6H_6$ , pet. ether.

Mitchell, Reid, *J. Am. Chem. Soc.*, 1931, **53**, 1881.

Soc. Française de Catalyse Généralisée, F.P., 701,579, (*Chem. Zentr.*, 1931, **II**, 1193).

Hoffmann, *Ber.*, 1882, **15**, 981.

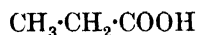
### Propionaphthone.

See Ethyl naphthyl Ketone.

### Propione.

See Diethyl Ketone.

### Propionic Acid



$C_3H_6O_2$

MW, 74

Colourless liq. with acrid odour. F.p.  $-19.7^\circ$ . B.p.  $141.35^\circ$ .  $D_4^{15}$  0.99874.  $n_D^{25}$  1.3859. Misc. in all proportions with  $H_2O$ . Heat of comb. 367.4 Cal. Mol. b.p. elevation  $35.1$ .  $k = 1.22 \times 10^{-5}$  at  $25^\circ$ . Most salts sol.  $H_2O$ .

*NH<sub>4</sub> salt*: very hygroscopic cryst. M.p.  $45^\circ$ . Sol.  $H_2O$ , EtOH, AcOH.

*K salt*: plates +  $1H_2O$  from EtOH.Aq. Becomes anhyd. at  $120^\circ$  and does not melt below  $300^\circ$ .

*Me ester*: see Methyl propionate.

*Et ester*:  $C_5H_{10}O_2$ . MW, 102. F.p.  $-72.6^\circ$ . B.p.  $99.1^\circ$ .  $D_4^{25}$  0.8830.  $n_D^{20}$  1.38385.

*Propyl ester*:  $C_6H_{12}O_2$ . MW, 116. B.p.  $122^\circ$ .  $D^{20}$  0.8809.

*Isopropyl ester*: b.p.  $109-11^\circ/750$  mm.  $D^0$  0.8931.

*Butyl ester*: see Butyl propionate.

*Isobutyl ester*: see Isobutyl propionate.

*n-Amyl ester*:  $C_8H_{16}O_2$ . MW, 144. F.p.  $-73.1^\circ$ . B.p.  $168-65^\circ$ .  $D_4^{15}$  0.8761.  $n_D^{15}$  1.4096.

*Isoamyl ester*: see Isoamyl propionate.

*tert.-Amyl ester*: see *tert.-Amyl propionate*.

*Decyl ester*:  $C_{13}H_{26}O_2$ . MW, 214. B.p.  $124^\circ/8$  mm.  $D_4^{20}$  0.8639.  $n_D^{20}$  1.42907.

*Glycerol mono-ester*: see Propionin.

*Glycerol di-ester*: dipropin.  $C_9H_{16}O_5$ . MW, 204. B.p.  $170-3^\circ/10$  mm.

*Glycerol tri-ester*: tripropin.  $C_{12}H_{20}O_6$ . MW, 260. B.p.  $177-82^\circ/20$  mm.,  $130-2^\circ/3$  mm.  $D_{18}^{100}$  1.100.  $n_D^{19}$  1.43175.

*Phenyl ester*:  $C_9H_{10}O_2$ . MW, 150. Prisms. M.p.  $20^\circ$ . B.p.  $211^\circ$ .

*Benzyl ester*:  $C_{10}H_{12}O_2$ . MW, 164. B.p.  $222^\circ$ .

*Chloride*: propionyl chloride.  $C_3H_5OCl$ . MW, 92.5. B.p.  $80^\circ$ .  $D_4^{20}$  1.0646.  $n_D^{20}$  1.40507.

*Bromide*: propionyl bromide.  $C_3H_5OBr$ . MW, 137. B.p.  $103-103.6^\circ/770$  mm.  $D^{16.4}$  1.5210.  $n_D^{19.4}$  1.45783.

*Amide*: see Propionamide.

*Anhydride*:  $C_6H_{10}O_3$ . MW, 130. B.p.  $168.4-168.8^\circ/780$  mm.,  $67.5^\circ/18$  mm.  $D^{15}$  1.0169.

*Nitrile*: propionitrile, ethyl cyanide.  $C_3H_5N$ . MW, 55. Liq. with ethereal odour. F.p.  $-103.5^\circ$ . B.p.  $97^\circ$ .  $D_4^{25}$  0.7770.  $n_D^{25}$  1.3659. Part misc. with  $H_2O$ . Mol. b.p. elevation  $22.6$ . Heat of comb.  $C_p$  446.7 Cal. Na, K  $\rightarrow$  di- and tri-molecular comps. AcOH  $\rightarrow$  *N*-acetylpropionamide. Extremely poisonous.

*Hydrazide*: cryst. M.p.  $40^\circ$ . B.p.  $130^\circ/16$  mm. Sol.  $H_2O$ . Spar. sol.  $Et_2O$ .

**Anilide**: propionanilide. Plates from EtOH.Aq., Et<sub>2</sub>O, or C<sub>6</sub>H<sub>6</sub>. M.p. 105–6°. Mod. sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O. Heat of comb. C<sub>p</sub> 1168.0 Cal.

**o-Nitroanilide**: lemon-yellow cryst. from EtOH. M.p. 63°. Sol. boiling H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

**p-Nitroanilide**: yellowish-brown plates from Ac<sub>2</sub>O. M.p. 182°.

**o-Toluidide**: cryst. M.p. 18°. B.p. 144–5°/11 mm.

**m-Toluidide**: needles from Et<sub>2</sub>O. M.p. 81°. Sol. EtOH, Et<sub>2</sub>O, ligroin.

**p-Toluidide**: plates from C<sub>6</sub>H<sub>6</sub>. M.p. 126°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold H<sub>2</sub>O, ligroin.

**1-Naphthylamide**: cryst. from EtOH. M.p. 116°.

Hardy, *J. Chem. Soc.*, 1936, 361, 365.

du Pont, U.S.P., 2,015,065, (*Chem. Zentr.*, 1936, I, 1310); U.S.P., 1,979,717, (*Chem. Abstracts*, 1935, 29, 181).

I.G., U.S.P., 2,005,183, (*Chem. Abstracts*, 1935, 29, 5125).

Werkmann, Rayburn, Hixon, U.S.P., 1,991,993, (*Chem. Abstracts*, 1935, 29, 2298).

Timmermans, Hennaut-Roland, *J. chim. phys.*, 1930, 27, 401.

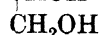
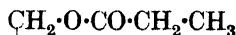
Pierre, Puchot, *Ann. chim.*, 1873, 28, 75.

Auger, *Compt. rend.*, 1907, 145, 1289.

Hanriot, Bouveault, *Bull. soc. chim.*, 1889, 1, 171.

Simons, *J. Am. Chem. Soc.*, 1926, 48, 1993.

**Propionin** (*Monopropin*, *glycerol α-propionate*)



C<sub>6</sub>H<sub>12</sub>O<sub>4</sub> MW, 148

B.p. 132–4°/3 mm. D<sub>4</sub><sup>20</sup> 1.1537.

**Diacetyl**: C<sub>10</sub>H<sub>16</sub>O<sub>6</sub>. MW, 232. B.p. 150°/12 mm.

Eastman Kodak, U.S.P., 2,005,371, (*Chem. Zentr.*, 1935, II, 2446).

Schuetz, Hale, *J. Am. Chem. Soc.*, 1930, 52, 1979.

Abderhalden, Eichwald, *Ber.*, 1914, 47, 1859.

**Propionitrile**.

See under Propionic Acid.

**Propionoin**.

See Diethylketol.

**Propionylacetamide**.

See Acetylpropionamide.

**Propionylacetic Acid** (2-Keto-*n*-valeric acid)



C<sub>5</sub>H<sub>8</sub>O<sub>3</sub> MW, 116

*Et ester*: C<sub>7</sub>H<sub>12</sub>O<sub>3</sub>. MW, 144. B.p. 191°, 75–8°/9 mm. Alc. FeCl<sub>3</sub> → red. col. Forms bisulphite comp.

*Nitrile*: C<sub>5</sub>H<sub>7</sub>ON. MW, 97. B.p. 164–5°. D<sub>20</sub> 0.976. Insol. H<sub>2</sub>O.

*Cu deriv.*: green cryst. M.p. 144–5°.

Décombe, *Ann. chim.*, 1932, 18, 81.

I.C.I., F.P., 666,703, (*Chem. Abstracts*, 1930, 24, 1120).

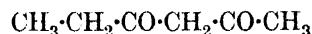
Breckport, *Chem. Abstracts*, 1924, 18, 816.

Wahl, *Compt. rend.*, 1911, 152, 97.

Dupont, *Compt. rend.*, 1909, 148, 1524.

Reymenant, *Chem. Zentr.*, 1901, I, 95.

**Propionylacetone** (*Acetylpropionylmethane*, *hexandione-2:4*, *2:4-diketohexane*)



C<sub>6</sub>H<sub>10</sub>O<sub>2</sub> MW, 114

Oil. B.p. 158°. D<sub>20</sub> 0.959. n<sub>D</sub><sup>20</sup> 1.4516.

*Cu deriv.*: blue needles. M.p. 197–8° (179°).

Powell, Seymour, *J. Am. Chem. Soc.*, 1931, 53, 1049.

Fittig, *Ann.*, 1907, 353, 24.

Claisen, Ehrhardt, *Ber.*, 1889, 22, 1014.

**Propionylanisole**.

See under 2-, and 4-Hydroxypropiofenone.

**Propionylbenzene**.

See Propiofenone.

**2-Propionylbenzoic Acid**.

See Propiofenone-*o*-carboxylic Acid.

**3-Propionyl-sec.-*n*-butyl Alcohol**.

See 3-Methyl-2-hexanolone-4.

**4-Propionyl-1-butylene**.

See 1-Heptenone-5.

**2-Propionyl-2-butylene**.

See 3-Methyl-2-hexenone-4.

**3-Propionylbutyric Acid**.

See 4-Keto-*n*-heptylic Acid.

**Propionylbutyryl**.

See Heptandione-3:4.

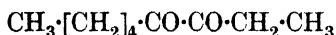
**Propionylbutyrylmethane**.

See Octandione-3:5.

**5-Propionyl-*n*-caproic Acid**.

See 6-Ketopelargonic Acid.

**Propionylcaproyl** (*Nonandione-3 : 4*, ethyl *n*-amyl diketone, 3 : 4-diketnononane)



$\text{C}_9\text{H}_{16}\text{O}_2$  MW, 156

B.p. 77–80°/10 mm.  $D_4^{20}$  0.927.

3-*Oxime*: needles. M.p. 33–4°. B.p. 131–2°/9 mm. Spar. sol. most org. solvents.

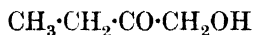
Dioxime: needles from boiling  $\text{C}_6\text{H}_6$ . M.p. 158°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. pet. ether. Sublimes.

Locquin, *Bull. soc. chim.*, 1904, 31, 1176.

### 7-Propionylcaprylic Acid.

See 8-Ketoundecylic Acid.

**Propionylcarbinol** (*Ethylketol*, 2-keto-*n*-butyl alcohol, hydroxymethyl ethyl ketone, 1-butanolone-2)



$\text{C}_4\text{H}_8\text{O}_2$  MW, 88

Colourless liq. B.p. 160°. Misc. with  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O.  $D_4^{15}$  1.0365.  $n_D^{14.5}$  1.4315. Reduces Fehling's. Acid  $\text{KMnO}_4 \rightarrow$  propionic acid. Forms bisulphite comp.

Formyl:  $\text{C}_5\text{H}_8\text{O}_3$ . MW, 116. B.p. 176–8°.  $D_4^{17}$  1.0946.  $n_D^{17}$  1.424. Semicarbazone: cryst. from  $\text{CHCl}_3$  or  $\text{C}_6\text{H}_6$ . M.p. 115°.

Me ether:  $\text{C}_5\text{H}_{10}\text{O}_2$ . MW, 102. B.p. 130–1°/729 mm. Phenylhydrazone: yellow oil. B.p. 170°/18 mm.

Et ether:  $\text{C}_6\text{H}_{12}\text{O}_2$ . MW, 116. Colourless oil turning yellow in air. B.p. 146°, 55°/24 mm.  $D_4^{16}$  0.914. Semicarbazone: m.p. 87°.

Isobutyl ether:  $\text{C}_8\text{H}_{16}\text{O}_2$ . MW, 144. B.p. 68–9°/13 mm. Semicarbazone: m.p. 72°.

Oxime: cryst. from  $\text{CHCl}_3$ . M.p. 60–1°.

Semicarbazone: cryst. from EtOH. M.p. 136–8°. Spar. sol.  $\text{H}_2\text{O}$ .

Sommelet, *Bull. soc. chim.*, 1911, 9, 35.

Kling, *Compt. rend.*, 1905, 140, 1345.

### Propionylcresol.

See Hydroxymethylpropiophenone.

### Propionyl cyanide.

See under 1-Ketobutyric Acid.

### Propionylcyclohexane.

See Hexahydropropiophenone.

### Propionylethylene.

See Ethyl vinyl Ketone.

### Propionylformic Acid.

See 1-Ketobutyric Acid.

### 2-Propionylheptane.

See 4-Methylnonanone-3.

### 6-Propionyl-*n*-heptylic Acid.

See 7-Ketocaproic Acid.

### Propionyl hydroperoxide.

See Perpropionic Acid.

### 1-Propionylisobutylene.

See 2-Methyl-2-hexenone-4.

### Propionynaphthalene.

See Ethyl naphthyl Ketone.

### 2-Propionyl-1-naphthol.

See Ethyl 1-hydroxy-2-naphthyl Ketone.

### *p*-Propionylphenetole.

See under 4-Hydroxypropiophenone.

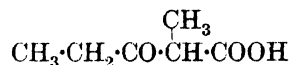
### Propionylphenol.

See 2-, and 4-Hydroxypropiophenone.

### 3-Propionylpropanol-1.

See 1-Hexanolone-4.

**1-Propionylpropionic Acid** (1-Methylpropionylacetic acid, 2-keto-1-methyl-*n*-valeric acid)



$\text{C}_6\text{H}_{10}\text{O}_3$  MW, 130

Me ester:  $\text{C}_7\text{H}_{12}\text{O}_3$ . MW, 144. Liq. with aromatic odour. B.p. 187°, 80°/12 mm.

Et ester:  $\text{C}_8\text{H}_{14}\text{O}_3$ . MW, 158. Oil. B.p. 199°, 87–8°/16 mm.  $D_4^{15}$  0.9827. Forms Na deriv. Red.  $\rightarrow$  2-hydroxy-1-methyl-*n*-valeric acid.  $\text{NaOH} \rightarrow$  diethyl ketone.  $\text{C}_6\text{H}_5 \cdot \text{NH} \cdot \text{NH}_2 \rightarrow$  4-methyl-3-ethyl-1-phenylpyrazolone-5. Di-Et acetal: b.p. 223–6°.

Isobutyl ester:  $\text{C}_{10}\text{H}_{18}\text{O}_3$ . MW, 186. B.p. 99–100°/11 mm.

Amide:  $\text{C}_6\text{H}_{11}\text{O}_2\text{N}$ . MW, 129. Hygroscopic cryst. M.p. 82°. Sol.  $\text{H}_2\text{O}$ .

Nitrile:  $\text{C}_6\text{H}_9\text{ON}$ . MW, 111. Oil with sweet odour. B.p. 193.5°.  $D_4^{20}$  0.9728.

Schroeter, *Ber.*, 1916, 49, 2719.

Pingel, *Ann.*, 1888, 245, 84.

Hantzsch, Wohlbrück, *Ber.*, 1887, 20, 1320.

### 2-Propionylpropionic Acid.

See 3-Keto-*n*-caproic Acid.

### 3-Propionyl-*n*-propyl Alcohol.

See 1-Hexanolone-4.

### Propionylpropylene.

See 1-Hexenone-4, 2-Hexenone-4, and 2-Methyl-1-pentenone-3.

### $\beta$ -Propionylstyrene.

See Ethyl styryl Ketone.

### Propionylthiophene.

See Propiothienone.

### Propionyltoluene.

See Ethyl tolyl Ketone.

### 4-Propionyl-*n*-valeric Acid.

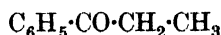
See 5-Keto-*n*-caprylic Acid.

### Propionylvaleryl.

See Octandione-3 : 4.



**Propiophenone** (*Ethyl phenyl ketone, propionylbenzene*)



$\text{C}_9\text{H}_{10}\text{O}$  MW, 134

Plates. M.p. 19–20°. B.p. 218°/764 mm., 115–20°/21 mm., 75°/3 mm.  $D_{25}^{20}$  1.0087.  $n_D^{20}$  1.534.  $\text{CrO}_3 \rightarrow$  benzoic and acetic acids. Na in EtOH  $\rightarrow$  ethylphenylcarbinol. Zn + HCl or H (+ Ni)  $\rightarrow$  propylbenzene.

*Oxime*: plates from pet. ether. M.p. 53–4°. B.p. 245–6° (decomp.), 165°/38 mm.

*2:4-Dinitrophenylhydrazones*: red leaflets from EtOH. M.p. 187–9°.

*Semicarbazone*: needles from EtOH. M.p. 182 (173°). Spar. sol. EtOH, Et<sub>2</sub>O.

*Di-Me acetal*:  $\text{C}_{11}\text{H}_{16}\text{O}_2$ . MW, 180. Oil. B.p. 206–8°, 92–3°/18 mm.  $D_4^{15}$  0.9888.

*Imide*: b.p. 102°/13 mm.

*Azine*: yellow needles. M.p. 79–80°. Sol. EtOH.

*Anil*: pale yellow needles from hexane. M.p. 50°. B.p. 169°/11 mm. Spar. sol. pet. ether.

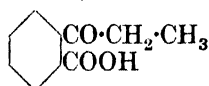
Thompson, Stevens, *J. Chem. Soc.*, 1932, 2611.

Shriner, Turner, *J. Am. Chem. Soc.*, 1930, 52, 1267.

Straus, Berkov, *Ann.*, 1913, 401, 140.

Auwers, *Ber.*, 1912, 45, 996.

**Propiophenone-*o*-carboxylic Acid** (*2-Propionylbenzoic acid*)



$\text{C}_{10}\text{H}_{10}\text{O}_3$  MW, 178

Needles from EtOH.Aq., or  $\text{C}_6\text{H}_6$ . M.p. 93° (97°). NaHg  $\rightarrow$  3-ethylphthalide.

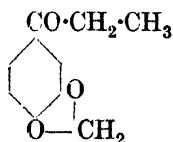
*Me ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_3$ . MW, 192. B.p. 157–8°/19 mm.  $D_4^{16.4}$  1.1274.  $n_D^{16.4}$  1.5197.

*Amide*:  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{N}$ . MW, 177. Needles from  $\text{H}_2\text{O}$ . Sinters at 150°. M.p. 159°. Spar. sol.  $\text{CHCl}_3$ , ligroin,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ .

*Anilide*: cryst. M.p. 160°. Insol.  $\text{H}_2\text{O}$ .

Auwers, Heinze, *Ber.*, 1919, 52, 590.

**Propiopiperone** (*3:4-Methylenedioxypropio-phenone*)



$\text{C}_{10}\text{H}_{10}\text{O}_3$  MW, 178

Needles. M.p. 39°. B.p. 153–4°/13 mm.  $D^{20}$  1.210.

*Oxime*: prisms. M.p. 104°.

*Semicarbazone*: m.p. 187–8°.

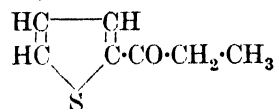
*Phenylhydrazone*: yellow needles. M.p. 97°.

Foulds, Robinson, *J. Chem. Soc.*, 1914, 105, 1972.

Schimmel, *Chem. Zentr.*, 1905, I, 1470.

Wallach, Pond, *Ber.*, 1895, 28, 2719.

**Propiothienone** (*2-Propionylthiophene, ethyl 1-thienyl ketone*)



$\text{C}_7\text{H}_8\text{OS}$  MW, 140

Pale yellow oil. B.p. 228°, 100–1°/11 mm. Ox.  $\rightarrow$  thiophene-2-carboxylic acid.

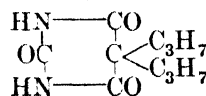
*Oxime*: plates. M.p. 55–6°.

*Semicarbazone*: plates from  $\text{C}_6\text{H}_6$ . M.p. 167°. Sol. hot  $\text{H}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. Et<sub>2</sub>O. Insol. pet. ether.

Steinkopf, Schubart, *Ann.*, 1921, 424, 8.

Thomas, Couderc, *Bull. soc. chim.*, 1918, 23, 288.

**Proponal** (*5:5-Dipropylbarbituric acid*)



$\text{C}_{10}\text{H}_{16}\text{O}_3\text{N}_2$  MW, 212

Colourless plates from  $\text{H}_2\text{O}$ . M.p. 146° (166°). Aq. sol. has bitter taste. Sol. 1640 parts  $\text{H}_2\text{O}$  at 20°, 70 parts at 100°. Very sol. EtOH, Et<sub>2</sub>O, AcOEt,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , alkalis. Forms mono-Na salt. Hypnotic.

*Quinine comp.*: needles. M.p. 127–8°. Very sol. EtOH,  $\text{Me}_2\text{CO}$ . Sol. Et<sub>2</sub>O,  $\text{CHCl}_3$ , hot  $\text{H}_2\text{O}$ , hot  $\text{C}_6\text{H}_6$ , hot ligroin.

Itallie, Steenhauer, *Pharm. Weekblad*, 1930, 67, 977, (*Chem. Zentr.*, 1931, I, 823).

Merck, D.R.P., 249,908, (*Chem. Zentr.*, 1912, II, 777); D.R.P., 146,496, (*Chem. Zentr.*, 1903, II, 1483).

Conrad, *Ann.*, 1905, 340, 321.

Fischer, Diltthey, *Ann.*, 1904, 335, 344.

**N-Propylacetamide.**

See under Propylamine.

**Propylacetanilide.**

See under Aminopropylbenzene.

**1-Propylacetacetic Acid.**

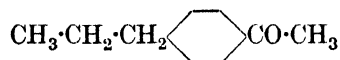
See 1-Aceto-*n*-valeric Acid.

**Propylacetonylcarbinol.**

See 4-Heptanolone-2.

**Propyl acetonyl Ether.**

See under Hydroxyacetone.

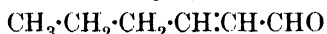
**p-Propylacetophenone** (p-Acetopropylbenzene) $\text{C}_{11}\text{H}_{14}\text{O}$  MW, 162Oil. B.p. 252°.  $D_4^{16}$  0.9785. Ox.  $\longrightarrow$  p-propylbenzoic acid.

Oxime: plates from pet. ether. M.p. 43–4°.

Phenylhydrazone: yellowish-white hexagonal plates from pet. ether. M.p. 92°. Very sol. warm pet. ether. Very unstable.

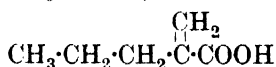
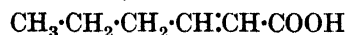
Widman, *Ber.*, 1888, 21, 2224.**n-Propylacetylene.**

See 1-Pentene.

**2-Propylacrolein** (2-Hexenal-1, 1-hexylene aldehyde, hexenic aldehyde) $\text{C}_6\text{H}_{10}\text{O}$  MW, 98Constituent of leaves of green plants. Colourless oil. B.p. 150°, 49.5°/18.5 mm.  $D_4^{13}$  0.861.  $n_D^{13}$  1.4470.

Semicarbazone: m.p. 175–6°.

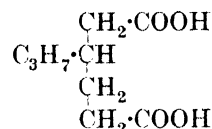
p-Nitrophenylhydrazone: m.p. 139°.

Delaby, Guillot-Allègre, *Bull. soc. chim.*, 1933, 53, 301.Curtius, Franzen, *Ann.*, 1914, 404, 93; 1912, 390, 89.**1-Propylacrylic Acid** ( $\alpha$ -Amylene- $\beta$ -carboxylic acid, 1-methylene-n-valeric acid, 1-pentene-2-carboxylic acid) $\text{C}_6\text{H}_{10}\text{O}_2$  MW, 114Prisms. M.p. 24.4°. B.p. 213°/750 mm.  $D_4^{25}$  0.9812. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ . Heat of comb.  $\text{C}_r$  795.7 Cal.  $k = 0.97 \times 10^{-5}$  at 25°. Gives spar. sol. Ca salt, cryst. Ag salt.Et ester:  $\text{C}_8\text{H}_{14}\text{O}_2$ . MW, 142. B.p. 167–8°.Lieben, Zeisel, *Monatsh.*, 1883, 4, 46.**2-Propylacrylic Acid** (1-Hexenic acid, 1:2-hexenoic acid,  $\alpha$ -amylene- $\alpha$ -carboxylic acid, 1-pentene-1-carboxylic acid, butylideneacetic acid, isohydrosorbic acid) $\text{C}_6\text{H}_{10}\text{O}_2$  MW, 114Needles from  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . M.p. 33°. B.p. 216–17°, 118°/19 mm.  $k = 1.98 \times 10^{-5}$  at25°. Br  $\longrightarrow$  1:2-dibromocaproic acid. HBr  $\longrightarrow$  2-bromocaproic acid.  $\text{AgClO}_3 \longrightarrow$  1:2-dihydroxycaproic acid.Et ester:  $\text{C}_8\text{H}_{14}\text{O}_2$ . MW, 142. B.p. 174–5°, 80°/22 mm.  $D_4^{20}$  0.8986.  $n_D^{20}$  1.4348.Chloride:  $\text{C}_6\text{H}_9\text{OCl}$ . MW, 132.5. B.p. 70°/23 mm.

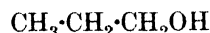
Anilide: needles. M.p. 109–10°.

Fittig, Baker, *Ann.*, 1894, 283, 118.Goldberg, Linstead, *J. Chem. Soc.*, 1928, 2351.Boxer, Linstead, *J. Chem. Soc.*, 1931, 740.Delaby, Guillot-Allègre, *Compt. rend.*, 1931, 192, 1467.**1-Propyladipic Acid.**

See Heptane-1:4-dicarboxylic Acid.

**2-Propyladipic Acid** $\text{C}_9\text{H}_{16}\text{O}_4$  MW, 188

Sinters at 45°. M.p. 49°. B.p. 184–6°/0.1 mm.

Dinitrile:  $\text{C}_9\text{H}_{14}\text{N}_2$ . MW, 150. B.p. 174–6°/12 mm.v. Braun, Keller, Weissbach, *Ann.*, 1931, 490, 182.**Propyl Alcohol (Propanol-1)** $\text{C}_3\text{H}_8\text{O}$  MW, 60B.p. 97.4°, 79–80°/374.6 mm., 65.94°/198.8 mm., 49.92°/90 mm., 30.35°/28.5 mm.  $D_4^{15}$  0.80753,  $D_4^{20}$  0.8035,  $D_4^{25}$  0.7993.  $n_D^{20}$  1.38499,  $n_D^{25}$  1.3833. Misc. with  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Crit. temp. 263.7°. KOH at 240–50°  $\longrightarrow$  propionic acid + propylene. Br  $\longrightarrow$  1:1-dibromopropionaldehyde + propyl bromide. I + KOH  $\longrightarrow$  iodoform.

Nitrite: see Propyl nitrite.

Nitrate: see Propyl nitrate.

Phosphate: see under Phosphoric Acid.

Acetyl: propyl acetate. B.p. 101.67°.  $D_4^{20}$  0.88630,  $D_4^{40}$  0.86390.  $n_D^{20}$  1.38422.Benzoyl: propyl benzoate. F.p. –51.6°. B.p. 230.6–230.9°.  $D_4^{15}$  1.0274.  $n_D^{15}$  1.50139.Heat of comb.  $\text{C}_r$  1255.010 Cal.

3:5-Dinitrobenzoyl: m.p. 73°.

Acid phthalate: m.p. 54–5°.

Phenylurethane: m.p. 58°.

p-Nitrophenylurethane: m.p. 115°.

1-Naphthylurethane : m.p. 105°.

Dreyfus, U.S.P., 1,996,101, (*Chem. Abstracts*, 1935, **29**, 3349).

du Pont, U.S.P., 2,014,740, (*Chem. Abstracts*, 1935, **29**, 7342).

Schüpphaus, *J. Am. Chem. Soc.*, 1892, **14**, 53.

Crismer, *Chem. Zentr.*, 1904, **I**, 1480.

Shriner, Cox, *J. Am. Chem. Soc.*, 1931, **53**, 1602.

### Propylallene.

See 1 : 2-Hexadiene.

### 3-Propylallyl Alcohol.

See 2-Hexenol-1.

### Propylallylamine



$\text{C}_6\text{H}_{13}\text{N}$  MW, 99

B.p. 110–14°.  $D^{15}_4$  0.7708. Sol. 15–20 parts  $\text{H}_2\text{O}$ .

Liebermann, Paal, *Ber.*, 1883, **16**, (I), 526.

### Propylallylcarbinol.

See 1-Heptenol-4.

### Propyl allyl Ether



$\text{C}_6\text{H}_{12}\text{O}$  MW, 100

B.p. 90–2°.  $D^{20}_4$  0.7764.

Deulofeu, *Chem. Zentr.*, 1928, **II**, 2547.

Lippert, *Ann.*, 1893, **276**, 192.

### Propyl allyl Ketone.

See 1-Heptenone-4.

### Propylamine (1-Aminopropane)



$\text{C}_3\text{H}_7\text{N}$  MW, 59

B.p. 49°.  $D^{15}_4$  0.7330,  $D^{15}_4$  0.714.  $n^{15}_D$  1.39006,  $n^{25}_D$  1.3873. Heat of comb.  $C_p$  (vapour) 575.74 Cal.,  $C_p$  (liq.) 560.3 Cal.,  $C_v$  (liq.) 559.4 Cal.  $k = 4.7 \times 10^{-4}$  at 25°.

$B, \text{HCl}$  : m.p. 157–8°.

$B, \text{HAuCl}_4$  : m.p. 169°.

$B_2, \text{H}_2\text{PtCl}_6$  : m.p. 214°.

$B_2, \text{H}_2\text{PtBr}_6$  : red leaflets. M.p. 257–8° decomp.

*Picrate* : m.p. 135°.

*N-Acetyl* : *N*-propylacetamide.  $\text{C}_6\text{H}_{11}\text{ON}$ . MW, 101. B.p. 222–5°.  $B, \text{HCl}$  : needles. M.p. 47°.

*N-Benzoyl* : *N*-propylbenzamide. Cryst. from EtOH or  $\text{C}_6\text{H}_6$ . M.p. 84.5°. B.p. 294–5°/750 mm. slight decomp.

Neogi, Chowdhuri, *J. Chem. Soc.*, 1917, **111**, 902.

Rakshit, *J. Am. Chem. Soc.*, 1913, **35**, 445.

Hofmann, *Ber.*, 1882, **15**, 769.

Linnemann, *Ann.*, 1872, **161**, 44.

### 2-Propylaminoethyl Alcohol (*N*-2-Hydroxyethylpropylamine, propylethanolamine)



$\text{C}_5\text{H}_{13}\text{ON}$  MW, 103

Oil. B.p. 182°/746 mm.  $D^{20}_4$  0.9005.  $n^{20}_D$  1.4428. Sol.  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O. Strongly basic.

$B, \text{HAuCl}_4$  : plates from  $\text{H}_2\text{O}$ . M.p. 85°.

*Picrate* : yellowish-brown prisms from  $\text{H}_2\text{O}$ . M.p. 129°. Sol.  $\text{H}_2\text{O}$ , EtOH.

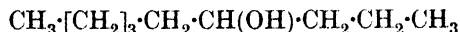
*Picrolonate* : brownish-red prisms from EtOH.Aq. M.p. 238° decomp.

Matthes, *Ann.*, 1901, **315**, 110.

### $\gamma$ -Propylaminopropylbenzene.

See under 3-Phenylpropylamine.

### Propyl-*n*-amylcarbinol (*Nonanol-4*)



$\text{C}_9\text{H}_{20}\text{O}$  MW, 144

B.p. 192–3°.  $D^{20}_4$  0.8282.  $n_D$ , 1.41971. Insol.  $\text{H}_2\text{O}$ .

*Acetyl* : b.p. 199–200°.  $D^{20}_4$  0.8282.

Pexters, *Chem. Zentr.*, 1907, **I**, 1398.

### *n*-Propyl-*tert*-amylcarbinol.

See 3 : 3-Dimethylheptanol-4.

### Propyl *n*-amyl Ketone (*Nonanone-4*)



$\text{C}_9\text{H}_{18}\text{O}$  MW, 142

B.p. 187–8°, 75–6°/20 mm., 70.5–71.5°/10 mm.  $D^{15}_4$  0.837.

*Semicarbazone* : cryst. from EtOH. M.p. 73–4° (rapid heat.) (67°, 145°).

*p-Nitrophenylhydrazone* : yellow cryst. from EtOH. M.p. 84–5°.

Karrer, Wettstein, Fröwis, Morf, *Helv.*

*Chim. Acta*, 1932, **15**, 231.

Bryant, Clemo, *J. Chem. Soc.*, 1931, 2080.

### N-Propylaniline



$\text{C}_9\text{H}_{13}\text{N}$  MW, 135

Pale yellow oil. B.p. 222°.  $D^{18}_4$  0.949.

*Hydrochloride*: needles. M.p. 150°.  
*Oxalate*: plates from H<sub>2</sub>O. M.p. 152°.

Voss, Blanke, *Ann.*, 1931, **485**, 280.

Hickinbottom, *J. Chem. Soc.*, 1930, 992.

### Propylaniline.

See Aminopropylbenzene.

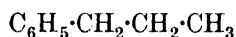
### Propylanisole.

See under Propylphenol.

### N-Propylbenzamide.

See under Propylamine.

### Propylbenzene (1-Phenylpropane)



C<sub>9</sub>H<sub>12</sub> MW, 120

M.p. -99.2°. B.p. 159.45°. D<sub>4</sub><sup>20</sup> 0.87864, D<sub>4</sub><sup>20</sup> 0.8617.  $n_D^{20}$  1.4925. Heat of comb. C<sub>p</sub> 1244.6 Cal. K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> + H<sub>2</sub>SO<sub>4</sub> → benzoic acid.

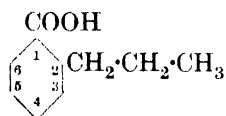
*Picrate*: yellow cryst. M.p. 103.5°.

Gilman, Catlin, *Organic Syntheses*, Collective Vol. I, 458.

Auwers, *Ann.*, 1919, **419**, 92.

Baril, Hauber, *J. Am. Chem. Soc.*, 1931, **53**, 1087.

### *o*-Propylbenzoic Acid



C<sub>10</sub>H<sub>12</sub>O<sub>2</sub> MW, 164

Leaflets from EtOH.Aq. M.p. 58°. B.p. 272°/739 mm.

*Et ester*: C<sub>12</sub>H<sub>16</sub>O<sub>2</sub>. MW, 192. B.p. 244-7°/785 mm. D<sub>15</sub><sup>15</sup> 1.003.

*Chloride*: C<sub>10</sub>H<sub>11</sub>OCl. MW, 182.5. Yellow liq. B.p. 236°.

*Amide*: C<sub>10</sub>H<sub>13</sub>ON. MW, 163. Needles from H<sub>2</sub>O. M.p. 127-8°. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

*Nitrile*: C<sub>10</sub>H<sub>11</sub>N. MW, 145. B.p. 227-9°/758 mm.

Gabriel, Michael, *Ber.*, 1878, **11**, 1014.

Gottlieb, *Ber.*, 1899, **32**, 961.

### *p*-Propylbenzoic Acid.

Prisms or leaflets from H<sub>2</sub>O. M.p. 141°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. boiling H<sub>2</sub>O. Volatile in steam. KMnO<sub>4</sub> → terephthalic acid.

*Nitrile*: b.p. 227°.

Widman, *Ber.*, 1889, **22**, 2278.

Francksen, *Ber.*, 1884, **17**, 1229.

### $\alpha$ -Propylbenzyl Alcohol.

See Propylphenylcarbinol.

**Propylbenzylcarbinol** (1-Phenylpentanol-2, 1-phenyl-sec.-*n*-amyl alcohol,  $\beta$ -hydroxy-*n*-amylbenzene)



C<sub>11</sub>H<sub>16</sub>O MW, 164

B.p. 127°/15 mm. D<sub>4</sub><sup>25</sup> 0.9579.  $n_D^{25}$  1.51017.

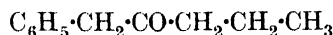
*Phenylurethane*: needles from pet. ether. M.p. 80°.

Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 151.

### Propylbenzylethylene.

See 1-Phenylhexene-2.

**Propyl benzyl Ketone** ( $\omega$ -Butyryltoluene,  $\beta$ -ketoamylbenzene, 1-phenylpentanone-2)



C<sub>11</sub>H<sub>14</sub>O MW, 162

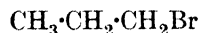
B.p. 244°. D<sub>4</sub><sup>0</sup> 0.984.

*Semicarbazone*: m.p. 84°.

Senderens, *Ann. chim.*, 1913, **28**, 321.

Ludlam, *J. Chem. Soc.*, 1902, **81**, 1189.

### Propyl bromide (1-Bromopropane)



C<sub>3</sub>H<sub>7</sub>Br MW, 123

B.p. 71°. Spar. sol. H<sub>2</sub>O. D<sub>4</sub><sup>20</sup> 1.3529.  $n_D^{20}$  1.43414. Heat of comb. C<sub>p</sub> 499.3 Cal. Heat with AlBr<sub>3</sub> → isopropyl bromide.

Karvonen, *Chem. Abstracts*, 1920, **14**, 2176.

Tseng, Hou, *J. Chinese Chem. Soc.*, 1934, **2**, 57.

Bodroux, *Compt. rend.*, 1915, **160**, 205.

Norris, *Am. Chem. J.*, 1907, **38**, 640.

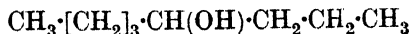
### 3-Propylbutanone.

See 3-Methylhexanone-2.

### 3-Propyl-sec.-*n*-butyl Alcohol.

See 3-Methylhexanol-2.

**Propyl-*n*-butylcarbinol** (Octanol-4, 4-hydroxyoctane)



C<sub>8</sub>H<sub>18</sub>O MW, 130

*d*l-.

B.p. 79°/16 mm. D<sub>4</sub><sup>22</sup> 0.818.  $[\alpha]_D^{22} + 0.74^\circ$ .

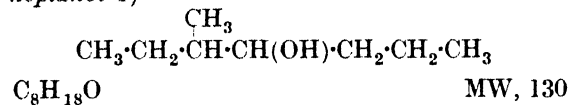
*dl*l-.

B.p. 71°/10 mm. D<sub>4</sub><sup>0</sup> 0.838.

Levene, Marker, *J. Biol. Chem.*, 1931, **91**, 405.

Bouveault, Locquin, *Bull. soc. chim.*, 1906, **35**, 644, 646.

**Propyl-sec.-n-butylcarbinol** (3-Methyl-heptanol-4)



$D^{22}_D$  0.8272.  $n^{25}_D$  1.4286.

Bridgman, *Chem. Zentr.*, 1933, II, 348.

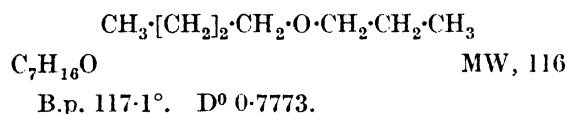
**Propyl-tert.-butylcarbinol.**

See 2 : 2-Dimethylhexanol-3.

**Propylbutylene.**

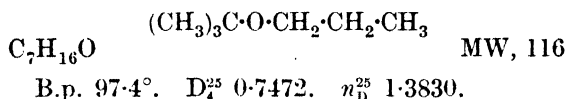
See 3-Methyl-1-hexene and 3-Methyl-2-hexene.

**Propyl n-butyl Ether**



Dobriner, *Ann.*, 1888, 243, 7.

**Propyl tert.-butyl Ether**

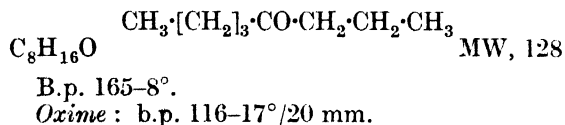


Norris, Rigby, *J. Am. Chem. Soc.*, 1932, 54, 2088.

**1-Propyl-2-butylethylene.**

See 4-Nonene.

**Propyl butyl Ketone** (4-Keto-octane, octanone-4)



Bouveault, Locquin, *Bull. soc. chim.*, 1906, 35, 648.

**2-Propylbutyraldehyde.**

See 2-Methylcaproic Aldehyde.

**2-Propylbutyric Acid.**

See 2-Methylcaproic Acid.

**Propylcarbamic Acid.**

Et ester, see Propylurethane : Amide, see Propylurea.

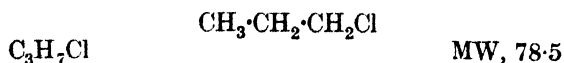
**Propylcarbylamine.**

See Propyl isocyanide.

**Propylcatechol.**

See 2 : 3-Dihydroxy-1-propylbenzene and 3 : 4-Dihydroxy-1-propylbenzene.

**Propyl chloride** (1-Chloropropane)



M.p. - 122.8°. B.p. 46.60°.  $D^{15}_4$  0.89694,  $D^{20}_4$  0.8910.  $n^{20}_D$  1.38838. Heat of comb.  $\text{C}_p$  (vapour) 492.38 Cal.

Norris, Taylor, *J. Am. Chem. Soc.*, 1924, 46, 756.

Dehn, Davis, *J. Am. Chem. Soc.*, 1907, 29, 1329.

**3-Propylcrotonaldehyde.**

See 1-Heptenal.

**2-Propylcrotonic Acid.**

See 2-Methyl-1-hexenic Acid.

**Propyl cyanide.**

See under n-Butyric Acid.

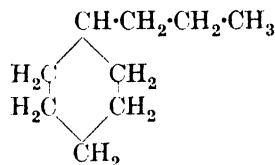
**Propylcyanoacetic Acid.**

See under Propylmalonic Acid.

**1-Propyl-1-cyanoisovaleric Acid.**

See under Propylisopropylmalonic Acid.

**Propylcyclohexane** (Hexahydropropylbenzene)



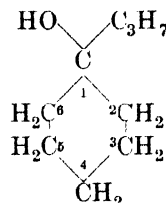
$\text{C}_9\text{H}_{18}$  MW, 126

Liq. with odour resembling petrol. B.p. 154.5-155.5°/756 mm.  $D^{20}_{400}$  0.7898.  $n^{21}_D$  1.437.

Eisenlohr, *Chem. Abstracts*, 1926, 20, 171.

Bourguet, *Bull. soc. chim.*, 1927, 41, 1475.

**1-Propylcyclohexanol** (1-Propylhexahydrophenol)



$\text{C}_9\text{H}_{18}\text{O}$  MW, 142

Liq. with odour resembling camphor. B.p. 180° decomp., 85°/20 mm.  $D^0$  0.945,  $D^{12}_4$  0.934.  $n^{12}_D$  1.468. Insol.  $\text{H}_2\text{O}$ .

Sabatier, Mailhe, *Bull. soc. chim.*, 1905, 33, 75.

**2-Propylcyclohexanol** (2-Propylhexahydrophenol).

Cis :

B.p. 84°/10 mm.  $D^{11}_4$  0.9247.  $n^{11}_D$  1.4688.

Isosvaleryl : b.p. 138-9°/18 mm.  $D^{11}_4$  0.914.  $n^{11}_D$  1.450.

*Acid succinate*: cryst. from AcOH.Aq. M.p. 31-2°.

*Benzoyl*: b.p. 177-8°/14 mm.  $D_4^{16}$  1.0262.  $n_D^{16}$  1.5150.

*Acid phthalate*: cryst. from EtOH.Aq. M.p. 107-8°.

*Phenylurethane*: cryst. from pet. ether. M.p. 97-97.5°.

*Trans*:

B.p. 90°/14 mm.  $D_4^{11}$  0.9160.  $n_D^{11}$  1.4668.

*Isovaleryl*: b.p. 129-30°/13 mm.  $D_4^{11}$  0.9131.  $n_D^{11}$  1.4490.

*Acid succinate*: m.p. 48-9°.

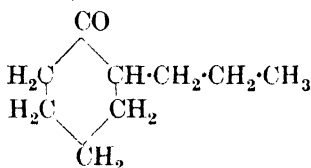
*Benzoyl*: b.p. 179-80°/15 mm.  $D_4^{16}$  1.0154.  $n_D^{16}$  1.513.

*Acid phthalate*: plates from EtOH.Aq. M.p. 120-1°.

*Phenylurethane*: m.p. 69-70°.

Vavon, Anziani, *Bull. soc. chim.*, 1927, 41, 1638.

## 2-Propylcyclohexanone



$C_9H_{16}O$

MW, 140

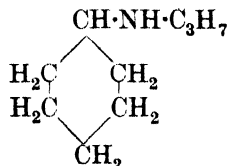
B.p. 198-9°/748 mm., 83-4°/13 mm.  $D_4^{13}$  0.9145.  $n_D^{13}$  1.4558.

*Oxime*: cryst. from EtOH.Aq. M.p. 67-8°.

*Semicarbazone*: cryst. from EtOH.Aq. M.p. 133.5-134° decomp.

See previous reference.

**N-Propylcyclohexylamine** (*Hexahydro-N-propylaniline*)



$C_9H_{19}N$

MW, 141

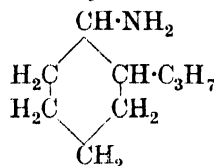
B.p. 185°.

*Hydrochloride*: m.p. 248-50°.

*Phenylurea deriv.*: needles from EtOH. M.p. 122-3°.

Skita, Keil, *Monatsh.*, 1929, 53 and 54, 759.

## 2-Propylcyclohexylamine



$C_9H_{19}N$

MW, 141

B.p. 60°/14 mm.  $D_4^{19}$  0.8752.

*N-Benzenesulphonyl*: m.p. 131-2°.

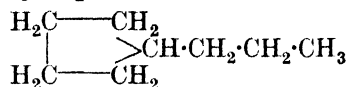
*Methiodide*: m.p. 208-10°.

v. Braun, Bayer, *Ber.*, 1925, 58, 390.

## Propyl cyclohexyl Ketone.

See Hexahydrobutyrophenone.

## Propylcyclopentane



$C_8H_{16}$

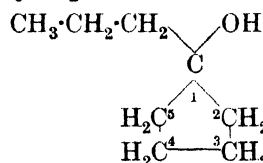
MW, 112

M.p. -120.3°. B.p. 131.3-131.5°, 126-8°/739 mm.  $D_4^{15}$  0.7814,  $D_4^{20}$  0.7772.  $n_D^{20}$  1.4266.

Chavanne, Becker, *Bull. soc. chim. Belg.*, 1927, 36, 591.

Zelinskii, Kazanskii, *Chem. Abstracts*, 1935, 29, 153.

## 1-Propylcyclopentanol



$C_8H_{16}O$

MW, 128

B.p. 175.2-175.7°.  $D_4^{15}$  0.9083,  $D_4^{20}$  0.9044.  $n_D^{20}$  1.4540.

*Allophanate*: m.p. 178°.

See first reference above.

## 2-Propylcyclopentanol.

*Cis*:

B.p. 79-80°/12 mm.  $D_4^9$  0.9165.  $n_D^9$  1.4600.

*Acid succinate*: m.p. 27-8°.

3:5-Dinitrobenzoyl: cryst. from EtOH. M.p. 70-1°.

*Acid phthalate*: needles. M.p. 95-6°.

*Phenylurethane*: m.p. 83-4°.

*Trans*:

B.p. 78-9°/10 mm.  $D_4^9$  0.9018.  $n_D^9$  1.4565.

*Acid succinate*: viscous oil.  $D_{16}^{16}$  1.0686.  $n_D^{16}$  1.4605.

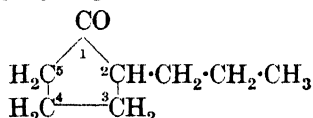
3:5-Dinitrobenzoyl: plates from EtOH. M.p. 30-1°.

*Acid phthalate*: cryst. from EtOH.Aq. M.p. 68°.

*Phenylurethane*: cryst. from EtOH.Aq. M.p. 61–2°.

Vavon, Flurer, *Bull. soc. chim.*, 1929, **45**, 756.

### 2-Propylcyclopentanone



$C_8H_{14}O$

MW, 126

B.p. 183.1–183.2°, 70.2°/15 mm., 59.4°/8 mm.  $D_4^{20}$  0.9017.  $n_D^{20}$  1.4429.

*Oxime*: b.p. 109–11°/9 mm.

*Semicarbazone*: cryst. from EtOH. M.p. 214° decomp.

Chiurdoglu, *Bull. soc. chim. Belg.*, 1934, **43**, 35.

Vavon, Flurer, *Bull. soc. chim.*, 1929, **45**, 754.

### 3-Propylcyclopentanone.

B.p. 190–1°.  $D_4^{20}$  0.9041.  $n_D^{20}$  1.4456.

*Oxime*: b.p. 121–2°/12 mm.

*Semicarbazone*: cryst. from MeOH. M.p. 178–9°.

v. Braun, Keller, Weissbach, *Ann.*, 1931, **490**, 181.

### Propyldichloroamine.

See *N*-Dichloropropylamine.

### $\alpha$ -Propyldiphenylmethane.

See 1:1-Diphenyl-*n*-butane.

### Propylene (*Propene*)



$C_3H_6$

MW, 42

Colourless gas. M.p. –185.2°. B.p. –47.8°/750 mm.  $n_{5876}^{20}$  1.001. Crit. temp. 92.1°. Liquefies under 7–8 atms. press. Heat of comb.  $C_p$  499.3 Cal. Abs. EtOH absorbs 12–13 vols. Absorbed quantitatively by solutions of mercuric nitrate and sulphate.

*Nitrosite*: prisms. M.p. 119–20°.  $Sn + HCl \rightarrow$  propylenediamine.

Ipat'ev, *Ber.*, 1934, **67**, 1061.

Goudet, Schenker, *Helv. Chim. Acta*, 1927, **10**, 135.

Rosenthal, U.S.P., 1,939,084, (*Chem. Abstracts*, 1934, **28**, 1364).

Maximoff, U.S.P., 1,870,859, (*Chem. Zentr.*, 1932, II, 3013).

Senderens, Aboulenc, *Chem. Zentr.*, 1936, I, 3818.

### Propylene bromide.

See 1:2-Dibromopropane.

### Propylene-carboxylic Acid.

See Crotonic Acid, Isocrotonic Acid, 1-Methylacrylic Acid, and Vinylacetic Acid.

### Propylene chloride.

See 1:2-Dichloropropane.

### Propylene chlorohydrin.

See 2-Chloropropyl Alcohol and 1-Chloroisopropyl Alcohol.

### Propylene chloriodide.

See 2-Chloro-1-iodopropane.

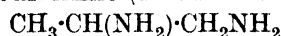
### Propylene cyanide.

See under Methylsuccinic Acid.

### Propylene-1:3-dialdehyde.

See Glutacondialdehyde.

### Propylenediamine (1:2-Diaminopropane)



$C_3H_{10}N_2$

MW, 74

*d*–.

B.p. 120.5°.  $D_4^{25}$  0.8584.  $[\alpha]_D^{25} + 29.78^\circ$ .

*Di-l-tartrate*: m.p. 143°.

*l*–.

B.p. 120.5°.  $D_4^{25}$  0.8588. Sol.  $H_2O$ ,  $CHCl_3$ . Insol.  $Et_2O$ .  $[\alpha]_D^{25} - 29.70^\circ$ . ( $[\alpha]_D^{24.3} - 20.957^\circ$ ).

*B,2HCl*: m.p. 240°.  $[\alpha]_D^{25} - 4.04^\circ$  in  $H_2O$ .

*Di-d-tartrate*: m.p. 143°.

*Picrate*: yellow cryst. M.p. 237°.

*dl*–.

B.p. 119–20°.  $D^{15}$  0.878. Forms hydrate with  $\frac{1}{2}H_2O$ .

*B,2HCl*: m.p. 220°.

1-*N-Di-Et*:  $C_7H_{18}N_2$ . MW, 130. B.p. 152°.

1:2-*N-Diacetyl*: needles from  $C_6H_6$ . M.p. 138–9°. B.p. 190°/18 mm. Very sol.  $H_2O$ , EtOH,  $CHCl_3$ . Spar. sol.  $C_6H_6$ . Insol.  $Et_2O$ , ligroin.

1:2-*N-Dipropionyl*: needles. M.p. 165°. B.p. 190°/56 mm.

1:2-*N-Dibenzoyl*: needles from  $C_6H_6$ . M.p. 192–3°.

*Dipicrate*: yellow needles from  $H_2O$ . M.p. 137°.

Windaus, Dorries, Jensen, *Ber.*, 1921, **54**, 2750.

I.G., D.R.P., 551,436, (*Chem. Zentr.*, 1932, II, 740).

Hofmann, *Ber.*, 1873, **6**, 308.

Tsugajew, Sokolow, *Ber.*, 1909, **42**, 56.

### Propylene dibromide.

See 1:2-Dibromopropane.

### Propylene-dicarboxylic Acid.

See Glutaconic Acid and Itaconic Acid.

### Propylene dichloride.

See 1:2-Dichloropropane.

**Propylene Glycol** (1 : 2-Dihydroxypropane,  $\alpha$ -propylene glycol)



$\text{C}_3\text{H}_8\text{O}_2$  MW, 76

*d*-.

B.p.  $95^\circ/15$  mm.  $[\alpha]_D^{18} + 13.71^\circ$  in  $\text{H}_2\text{O}$ .  
Readily racemises in  $\text{H}_2\text{O}$ .

*Dibutyl*: b.p.  $95\text{--}105^\circ/15$  mm.  $[\alpha]_D^{18} + 2.05^\circ$ .

*l*-.

B.p.  $85\text{--}91^\circ/12$  mm.  $[\alpha]_D^{20} - 9.8^\circ$  in  $\text{H}_2\text{O}$ .

*Di-phenylurethane*: m.p.  $146\text{--}7^\circ$ .  $[\alpha]_D^{20} + 13.3^\circ$  in EtOH.

*dl*-.

Viscous oil with sweet taste. B.p.  $188\text{--}9^\circ$ ,  $96\text{--}8^\circ/21$  mm.  $D_4^{20} 1.0403$ . Misc. with  $\text{H}_2\text{O}$  and EtOH in all proportions. Conc. HI  $\rightarrow$  isopropyl iodide.  $\text{ZnCl}_2$  or 50%  $\text{H}_2\text{SO}_4 \rightarrow$  propionaldehyde.  $\text{H}_2\text{O}_2 + \text{FeSO}_4 \rightarrow$  acetone + propionaldehyde.

*Diacetyl*: b.p.  $190\text{--}1^\circ/762$  mm.  $D_4^{20} 1.059$ .  $n_D^{20} 1.4173$ .

*Di-phenylurethane*: m.p.  $152.5\text{--}153.5^\circ$ .

*Dinitrite*: b.p.  $108\text{--}10^\circ$ . Insol.  $\text{H}_2\text{O}$ .

*Monopalmitate*: plates from EtOH. M.p.  $54.3^\circ$ .

*Dipalmitate*: plates from EtOH. M.p.  $68.8^\circ$ .

*Monostearate*: plates from EtOH. M.p.  $59.5^\circ$ .

*Distearate*: plates from EtOH. M.p.  $72.3^\circ$ .

1-*Me ether*:  $\text{C}_4\text{H}_{10}\text{O}_2$ . MW, 90. B.p.  $126\text{--}7^\circ$ .  $D_4^{20} 0.9260$ .  $n_D^{20} 1.4070$ . *Acetyl*: b.p.  $147^\circ/762$  mm.  $D_4^{20} 0.9709$ .  $n_D^{20} 1.4045$ .

1-*Et ether*:  $\text{C}_5\text{H}_{12}\text{O}_2$ . MW, 104. B.p.  $136^\circ$ .  $D_4^{20} 0.9028$ .  $n_D^{20} 1.4$ . *Acetyl*: b.p.  $158\text{--}60^\circ$ .  $D_4^{20} 0.9461$ .  $n_D^{20} 1.4097$ .

2-*Et ether*: b.p.  $140\text{--}1^\circ$ .  $D_4^{20} 0.9044$ .  $n_D^{20} 1.4122$ . Sol.  $\text{H}_2\text{O}$ .

Morley, Green, *J. Chem. Soc.*, 1885, 47, 132.  
Nef, *Ann.*, 1904, 335, 291.

Ochiai, Miyaki, *Biochem. Z.*, 1935, 282, 293.

Klebanskii, Dolgopol'skii, *Chem. Abstracts*, 1935, 29, 5814.

du Pont, U.S.P., 1,963,997, (*Chem. Zentr.*, 1934, II, 3314).

Matignon, Moureu, Dodé, *Chem. Zentr.*, 1933, II, 646.

Levene, Walti, *J. Biol. Chem.*, 1926, 68, 415.

Dewael, *Bull. soc. chim. Belg.*, 1930, 39, 395.

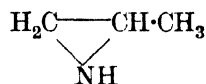
Abderhalden, Eichwald, *Ber.*, 1918, 51, 1319.

Howe, *Chem. Abstracts*, 1919, 13, 1843.

**$\beta$ -Propylene Glycol.**

See Trimethylene Glycol.

**Propyleneimine**



$\text{C}_3\text{H}_7\text{N}$

MW, 57

Oil. B.p.  $66\text{--}7^\circ/751$  mm.  $D^{16} 0.812$ . Fumes in air. Decom. in aq. or HCl sol. Evaporate with HCl  $\rightarrow$  2-chloroisopropylamine.

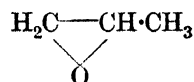
Gabriel, v. Hirsch, *Ber.*, 1896, 29, 2747.

Gabriel, Ohle, *Ber.*, 1917, 50, 815.

**Propylene iodohydrin.**

See 1-Iodoisopropyl Alcohol.

**Propylene oxide**



$\text{C}_3\text{H}_6\text{O}$

MW, 58

*dl*-.

Liq. with odour resembling ether. B.p.  $35^\circ$ .  $D^0 0.859$ . Misc. with  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ .  $\text{Al}_2\text{O}_3$  at  $250\text{--}60^\circ \rightarrow$  propionaldehyde + a little acetone.  $\text{Ag}_2\text{O} \rightarrow$  acetic acid.  $\text{NaHg} + \text{H}_2\text{O} \rightarrow$  isopropyl alcohol. Hot  $\text{H}_2\text{O} \rightarrow$  propylene glycol.  $\text{C}_2\text{H}_5\text{MgI}$  in  $\text{Et}_2\text{O} \rightarrow$  methylpropylcarbinol.

*d*-.

B.p.  $36.5\text{--}38^\circ$ .  $[\alpha]_D^{18} + 12.72^\circ$ . Part. racemises with hot  $\text{H}_2\text{O}$ .

*l*-.

$[\alpha]_D^{18} - 8.26^\circ$ . Part. racemises with hot  $\text{H}_2\text{O}$ .

I.G., E.P., 292,066, (*Chem. Abstracts*, 1929, 23, 1415).

Abderhalden, Eichwald, *Ber.*, 1918, 51, 1318.

Krassuski, *Chem. Zentr.*, 1902, II, 19.

Henry, *Chem. Zentr.*, 1903, II, 486.

**Propylene oxide carboxylic Acid.**

See Methylglycidic Acid.

**Propylene-tricarboxylic Acid.**

See Aconitic Acid and Isaconitic Acid.

**Propylethanolamine.**

See Propylaminoethyl Alcohol.

**Propyl *p*-ethoxyphenyl Ketone.**

See under *p*-Hydroxybutyrophenone.

**Propylethylene.**

See 1-Pentene.

**Propyl fluoride (1-Fluoropropane)**



$\text{C}_3\text{H}_7\text{F}$

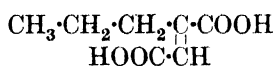
MW, 62



Gas. Liq. at  $-3^{\circ}$ . Burns with bright flame.

Meslans, *Ann. chim. phys.*, 1894, **1**, 363.  
du Pont, E.P., 406,284, (*Chem. Zentr.*, 1934, II, 132).

**Propylfumaric Acid** (1-Pentene-1 : 2-dicarboxylic acid,  $\alpha$ -amylene- $\alpha\beta$ -dicarboxylic acid, ethyl-mesaconic acid)



$\text{C}_7\text{H}_{10}\text{O}_4$  MW, 158

Leaflets. M.p.  $174-5^{\circ}$  ( $172.5-173^{\circ}$ ). Very sol. EtOH, Et<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>. Very spar. sol. C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Sol. 90 parts H<sub>2</sub>O at ord. temp.  $k = 9.3 \times 10^{-4}$  at  $25^{\circ}$ . CH<sub>3</sub>COCl at  $105-10^{\circ} \rightarrow$  ethylcitraconic anhydride.

*Et ester-amide*: C<sub>9</sub>H<sub>15</sub>O<sub>3</sub>N. MW, 185. Needles. M.p.  $78-9^{\circ}$ .

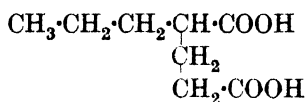
*Diamide*: C<sub>7</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>. MW, 156. M.p.  $214-15^{\circ}$  decomp.

Demarçay, *Ann. chim. phys.*, 1880, **20**, 489.

Walden, *Ber*, 1891, **24**, 2035.

Ssamenow, *Chem. Zentr.*, 1899, I, 783.

**1-Propylglutaric Acid** (Hexane-1 : 3-dicarboxylic acid)

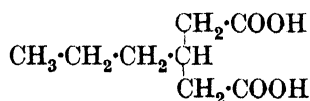


$\text{C}_8\text{H}_{14}\text{O}_4$  MW, 174

Cryst. from H<sub>2</sub>O. M.p.  $66-8^{\circ}$ .  $k = 5.86 \times 10^{-5}$  at  $24.4^{\circ}$ .

Mellor, *J. Chem. Soc.*, 1901, **79**, 129.

**2-Propylglutaric Acid**



$\text{C}_8\text{H}_{14}\text{O}_4$  MW, 174

Needles from HCl. M.p.  $52^{\circ}$ . Sol. H<sub>2</sub>O and most org. solvents.  $k$  (first)  $= 0.487 \times 10^{-4}$  at  $25^{\circ}$ : (second)  $= 4.11 \times 10^{-7}$  at  $25^{\circ}$ .

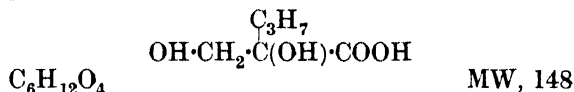
*Di-Et ester*: C<sub>12</sub>H<sub>22</sub>O<sub>4</sub>. MW, 230. B.p.  $132^{\circ}/10$  mm.

*Anhydride*: C<sub>8</sub>H<sub>12</sub>O<sub>3</sub>. MW, 156. Mobile oil. B.p.  $180^{\circ}/20$  mm.

*Monoanilide*: plates from C<sub>6</sub>H<sub>6</sub>. M.p.  $128^{\circ}$ .

Day, Thorpe, *J. Chem. Soc.*, 1920, **117**, 1471.

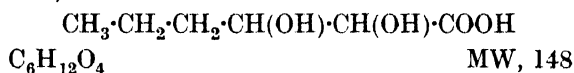
**1-Propylglyceric Acid** (1 : 2-Dihydroxypentane-2-carboxylic acid)



M.p.  $94-5^{\circ}$ . Sol. Et<sub>2</sub>O.

Ssamenow, *Chem. Zentr.*, 1899, I, 1071.

**2-Propylglyceric Acid** (1 : 2-Dihydroxycaproic acid, 1 : 2-dihydroxypentane-1-carboxylic acid)



Exists in two forms :

(i) Cryst. from AcOEt. M.p.  $108.5^{\circ}$ . Sol. hot H<sub>2</sub>O. Mod. sol. Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>.

*Phenylhydrazide*: m.p.  $141.5-142^{\circ}$ .

(ii) Cryst. from AcOEt. M.p.  $99.5^{\circ}$ . Sol. EtOH, hot H<sub>2</sub>O. Mod. sol. Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>.

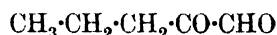
*Phenylhydrazide*: m.p.  $120-1^{\circ}$ .

Braun, *J. Am. Chem. Soc.*, 1930, **52**, 3190.

**1-Propylglycerol.**

*See* Hexantriol-1 : 2 : 3.

**Propylglyoxal** (1-Ketovaleraldehyde, 1-pentanalone-2)



$\text{C}_5\text{H}_8\text{O}_2$  MW, 100

Greenish liq. with strong odour. B.p.  $36^{\circ}/16$  mm. Reduces NH<sub>3</sub>.AgNO<sub>3</sub>. Colours Schiff's reagent. Polymerises.

*Disemicarbazone*: plates + 1AcOH from AcOH.Aq. M.p.  $250^{\circ}$ .

*Osazone*: needles from AcOH. M.p.  $105^{\circ}$ .

*Di-Me acetal*: C<sub>7</sub>H<sub>14</sub>O<sub>3</sub>. MW, 146. B.p.  $65-6^{\circ}/14$  mm. Reduces NH<sub>3</sub>.AgNO<sub>3</sub> but not Fehling's.

Blaise, *Compt. rend.*, 1922, **175**, 1216.

**Propylguaiacol.**

*See* Cœrulignol and under 2 : 3-Dihydroxy-1-propylbenzene.

**1-Propylguanidine**



$\text{C}_4\text{H}_{11}\text{N}_3$  MW, 101

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: m.p.  $220^{\circ}$  decomp.

*B<sub>3</sub>H<sub>3</sub>AuCl<sub>4</sub>*: red needles from EtOH. M.p.  $200^{\circ}$  decomp.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow prisms. M.p.  $195^{\circ}$  decomp.

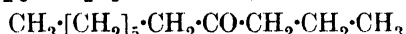
*Picrate*: yellow needles from EtOH. M.p. 177–8°.

Piovano, *Gazz. chim. ital.*, 1928, **58**, 245.

#### 4-Propyl-1-heptenol-4.

See Dipropylallylcarbinol.

#### Propyl heptyl Ketone (*Undecanone-4*)



$\text{C}_{11}\text{H}_{22}\text{O}$  MW, 170

Oil with characteristic fruity odour. M.p. 4–5°. B.p. 106–7°/13 mm.  $D_{25}^{25}$  0.8274.  $n_D^{25}$  1.4248.

*Oxime*: b.p. 141°/13 mm.

*Semicarbazone*: plates from EtOH. M.p. 54–6°.

v. Braun, Kröper, *Ber.*, 1929, **62**, 2882.

#### Propylhexahydrocresol.

See Methylpropylcyclohexanol.

#### Propylhexahydrophenol.

See Propylcyclohexanol.

#### Propylhexahydrotoluene.

See Methylpropylcyclohexane.

#### 3-Propylhexanol-3.

See Ethyldipropylcarbinol.

#### Propyl hexyl Ketone (*Decanone-4*)



$\text{C}_{10}\text{H}_{20}\text{O}$  MW, 156

M.p. – 9°. B.p. 206–7°, 87–9°/11 mm°.  $D_0^{30.5}$  0.824. Does not form bisulphite comp.

*Semicarbazone*: m.p. 51–2°.

Karrer, Shibata, Wettstein, Jacobowicz, *Helv. Chim. Acta*, 1930, **13**, 1300.

#### Propylhydrazine



$\text{C}_3\text{H}_{10}\text{N}_2$  MW, 74

B.p. 119°. Reduces  $\text{NH}_3 \cdot \text{AgNO}_3$  and Fehling's in the cold.

Stolle, Bernath, *J. prakt. Chem.*, 1904, **70**, 280.

#### Propylhydroquinone.

See 2 : 5-Dihydroxy-1-propylbenzene.

#### Propylhydroxylamine (*1-Hydroxylamino-propane*)



$\text{C}_3\text{H}_9\text{ON}$  MW, 75

Needles from  $\text{Et}_2\text{O}$ . M.p. 77° (about 46°). Very volatile. Spar. sol. ligroin. Reacts basic. Reduces Fehling's.

Pierron, *Bull. soc. chim.*, 1899, **21**, 784.

Kjellin, *Ber.*, 1897, **30**, 1892.

Dict. of Org. Comp.—III.

#### Propyl hydroxynaphthyl Ketone.

See Hydroxybutyronaphthone.

#### Propyl hydroxyphenyl Ketone.

See o-, m-, and p-Hydroxybutyphenone.

#### Propyl p-hydroxyphenyl sulphide.

See under Thiohydroquinone.

#### Propyl 1-hydroxypropyl Ketone.

See 3-Heptanolone-4.

#### Propyl hydroxytolyl Ketone.

See Hydroxy-methylbutyphenone.

#### Propylideneacetic Acid.

See 2-Ethylacrylic Acid.

#### Propylideneacetone.

See 3-Hexenone-2.

#### Propylideneaniline.

See under Propionaldehyde.

#### 2-Propylidenebutane.

See 3-Methyl-3-hexene.

#### Propylidenebutylene.

See 1 : 4-Heptadiene, 2 : 4-Heptadiene, and 3-Methyl-1 : 3-pentadiene.

#### 2-Propylidenebutyric Acid.

See 2-Methylhydrosorbic Acid.

#### Propylidene chloride.

See 1 : 1-Dichloropropane.

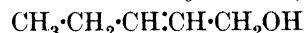
#### Propylidene chlorobromide.

See 1-Chloro-1-bromopropane.

#### $\alpha$ -Propylidenediphenylmethane.

See 1 : 1-Diphenylbutylene-1.

2-Propylidene-ethyl Alcohol (*2-Pentenol-1*, 3-ethylallyl alcohol,  $\alpha$ -butenylcarbinol)



$\text{C}_5\text{H}_{10}\text{O}$  MW, 86

Liq. B.p. 141–2° (138–9°).  $D_4^4$  0.8645,  $D_4^{20}$  0.8468.  $n_D^{20}$  1.4299.

*Acetyl*: b.p. 149–51°.  $D^{22}$  0.9019.  $n_D^{22}$  1.4219.

Bouis, *Ann. chim.*, 1928, **9**, 402.

Prévost, *Ann. chim.*, 1928, **10**, 113.

Delaby, *Ann. chim.*, 1923, **20**, 196.

#### Propylidene-ethylene.

See 1 : 2-Pentadiene.

#### 3-Propylideneisobutane.

See 2-Methyl-3-hexene.

#### 1-Propylideneisobutyric Acid.

See 1-Methylhydrosorbic Acid.

#### 2-Propylidenepropene.

See 2-Methyl-2-pentene.

#### 2-Propylidenepropionaldehyde.

See 1-Methyl-2-ethylacrolein.

#### 1-Propylidenepropionic Acid.

See 1-Methyl-2-ethylacrylic Acid.

#### 2-Propylidenepropionic Acid.

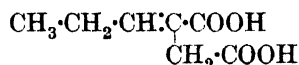
Hydrosorbic Acid, *q.v.*

#### Propylidenepropyl Alcohol.

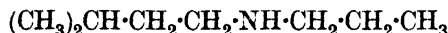
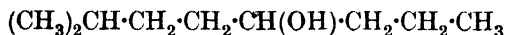
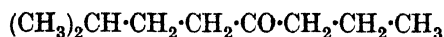
See 3-Hexenol-1 and 2-Methyl-2-pentenol-1.

**3-Propylidene-propylene.**

See 1 : 3-Hexadiene.

**Propylidenesuccinic Acid** (*Ethylitaconic acid, 2-pentene-1 : 2-dicarboxylic acid*) $\text{C}_7\text{H}_{10}\text{O}_4$  MW, 158Prisms from  $\text{H}_2\text{O}$ . M.p.  $164-5^\circ$  ( $163-7^\circ$  decomp.). Spar. sol.  $\text{CHCl}_3$ , cold  $\text{H}_2\text{O}$ . Very spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin.  $k = 3.56 \times 10^{-5}$  at  $25^\circ$ .Fichter, Probst, *Ann.*, 1910, **372**, 76.**Propyl iodide (1-Iodopropane)** $\text{C}_3\text{H}_7\text{I}$  MW, 170B.p.  $102.5^\circ$ .  $D_4^{20}$  1.7471.  $n_D^{20}$  1.50546. Heat of comb.  $C_p$  514.3 Cal.,  $C_v$  512.3 Cal.Adams, Voorhees, *J. Am. Chem. Soc.*, 1919, **41**, 797.Hirao, *J. Chem. Soc. Japan*, 1931, **52**, 269.**1-Propylisoamyl Alcohol.**

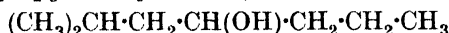
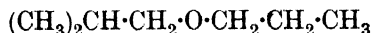
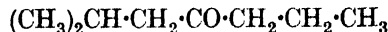
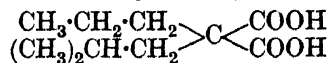
See Propylisobutylcarbinol.

**Propylisoamylamine** $\text{C}_8\text{H}_{19}\text{N}$  MW, 129Liq. B.p.  $148-9^\circ$  ( $141^\circ$ ).Sabatier, Mailhe, *Compt. rend.*, 1909, **148**, 900.Freundler, Juillard, *ibid.*, 290.**Propylisoamylcarbinol** (2-Methyloctanol-5, 1-propylisohexyl alcohol) $\text{C}_9\text{H}_{20}\text{O}$  MW, 144B.p.  $184-6^\circ$ .  $D_4^0$  0.8335,  $D_4^{16}$  0.8199.Douris, *Compt. rend.*, 1913, **157**, 57.**Propyl isoamyl Ketone** (2-Methyloctanone-5) $\text{C}_{10}\text{H}_{18}\text{O}$  MW, 142B.p.  $177-9^\circ$ .  $D_4^0$  0.8362,  $D_4^{21}$  0.8205.*Semicarbazone* : m.p.  $107^\circ$  ( $102^\circ$ ).Douris, *Compt. rend.*, 1913, **157**, 56.**Propylisobutylacetic Acid.**

See 1-Propylisocaproic Acid.

**1-Propylisobutyl Alcohol.**

See Propylisopropylcarbinol.

**Propylisobutylamine** $\text{C}_7\text{H}_{17}\text{N}$  MW, 115Liq. with odour resembling fusel-oil. B.p.  $123-5^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ .*B.HCl* : plates from  $\text{EtOH}-\text{Et}_2\text{O}$ . M.p.  $135^\circ$ .*Acid oxalate* : needles. M.p.  $224^\circ$ .*B.HAuCl\_4* : m.p.  $187-8^\circ$ . Very sol.  $\text{EtOH}$ .*B\_2H\_2PtCl\_6* : orange cryst. M.p.  $187-8^\circ$ . Sol.  $\text{EtOH}$ , hot  $\text{H}_2\text{O}$ .Paal, Heupel, *Ber.*, 1891, **24**, 3048.Pope, Read, *J. Chem. Soc.*, 1912, **101**, 522.**Propylisobutylcarbinol** (2-Methylheptanol-4, 1-propylisoamyl alcohol) $\text{C}_8\text{H}_{18}\text{O}$  MW, 130Somewhat viscous pleasant-smelling liq. B.p.  $164^\circ$  ( $160^\circ$ ).  $D^{20}$  0.8207.  $n_D$  1.42031. Sol. most org. solvents. Insol.  $\text{H}_2\text{O}$ .Clarke, *J. Am. Chem. Soc.*, 1909, **31**, 114.Muset, *Chem. Zentr.*, 1907, **I**, 1313.**Propyl isobutyl Ether** $\text{C}_7\text{H}_{16}\text{O}$  MW, 116B.p.  $106^\circ/720$  mm.  $D^{15}$  0.7549.  $n_{D_{461}}^{25.9}$  1.3852. Very spar. sol.  $\text{H}_2\text{O}$ .Henstock, *J. Chem. Soc.*, 1931, 371.**Propyl isobutyl Ketone** (2-Methylheptanone-4) $\text{C}_8\text{H}_{16}\text{O}$  MW, 128B.p.  $155^\circ/750$  mm.  $D_0^{22}$  0.813. Does not form bisulphite comp.*Semicarbazone* : m.p.  $123-4^\circ$ .Fournier, *Bull. soc. chim.*, 1910, **7**, 839.**Propylisobutylmalonic Acid** (2-Methylheptane-4 : 4-dicarboxylic acid) $\text{C}_{10}\text{H}_{18}\text{O}_4$  MW, 202Prisms or plates from  $\text{H}_2\text{O}$ . M.p.  $147-9^\circ$  decomp. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{C}_6\text{H}_6$ , cold  $\text{H}_2\text{O}$ .*Di-Et ester* :  $\text{C}_{14}\text{H}_{26}\text{O}_4$ . MW, 258. B.p.  $126^\circ/9.5$  mm.Fischer, Holzapfel, v. Gwinner, *Ber.*, 1912, **45**, 252.

**1-Propylisocaproic Acid** (2-Methylheptane-4-carboxylic acid, propylisobutylic acid)

$\text{C}_9\text{H}_{18}\text{O}_2$   $(\text{CH}_3)_2\text{CH}\cdot\text{CH}_2\cdot\overset{\text{C}_3\text{H}_7}{\text{CH}}\cdot\text{COOH}$  MW, 158

*d.*  
Oil. B.p. about 100°/0.5 mm.  $D^{20}_D$  0.8876.  
 $[\alpha]^{20}_D + 9.80^\circ$ .

*dl.*  
B.p. 125–7°/12 mm., 122°/8.5 mm.  $D^{20}_D$  0.8928. Sol. EtOH, Et<sub>2</sub>O. Very spar. sol. H<sub>2</sub>O.

Fischer, Holzapfel, v. Gwinner, *Ber.*, 1912, 45, 253.

**Propyl isocyanide** (Propylcarbylamine)

$\text{C}_4\text{H}_7\text{N}$   $\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{NC}$  MW, 69

B.p. 99–5°. Heat of comb.  $C_p$  638.9 Cal.

Guillemand, *Ann. chim. phys.*, 1908, 14, 412.

**1-Propylisohexyl Alcohol.**

See Propylisoamylcarbinol.

**Propylisopropylacetic Acid.**

See 2-Methylhexane-3-carboxylic Acid.

**Propylisopropylcarbinol** (2-Methylhexanol-3, 1-propylisobutyl alcohol)

$(\text{CH}_3)_2\text{CH}\cdot\text{CH}(\text{OH})\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_3$   
 $\text{C}_7\text{H}_{16}\text{O}$  MW, 116

*d.*  
B.p. 145–6°.  $D^{18.7}_D$  0.8266.  $[\alpha]^{20}_D + 21.25^\circ$ .  
Acid phthalate: m.p. 79–80°.  $[\alpha]_D + 8.36^\circ$  in EtOH.

*dl.*  
Pleasant-smelling liq. with burning taste. B.p. 141–2°/765 mm.  $D^{17}_D$  0.821.  $n_D$  1.41493. Insol. H<sub>2</sub>O.

Acid phthalate: m.p. 59–60°.

Muset, *Chem. Zentr.*, 1907, I, 1313.

Pickard, Kenyon, *J. Chem. Soc.*, 1912, 101, 633.

**Propyl isopropyl Ether**

$(\text{CH}_3)_2\text{CH}\cdot\text{O}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_3$   
 $\text{C}_6\text{H}_{14}\text{O}$  MW, 102

B.p. 83°.  $D^{20}_D$  0.7597,  $D^{12.5}_D$  0.7474.  $n^{21}_D$  1.376.

Bennett, Philip, *J. Chem. Soc.*, 1928, 1930.

Truchet, Graves, *Bull. soc. chim.*, 1932, 51, 686.

**Propyl isopropyl Ketone** (2-Methylhexanone-3)

$(\text{CH}_3)_2\text{CH}\cdot\text{CO}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_3$   
 $\text{C}_7\text{H}_{14}\text{O}$  MW, 114  
Oil with peppermint odour. B.p. 135–6° (129–30°).

Semicarbazone: m.p. 119°.

Meerwein, *Ann.*, 1919, 419, 138.

**Propylisopropylmalonic Acid** (2-Methylhexane-3:3-dicarboxylic acid)

$\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{C}(\text{COOH})_2$   
 $(\text{CH}_3)_2\text{CH}\cdot\text{C}(\text{COOH})_2$   
 $\text{C}_9\text{H}_{16}\text{O}_4$  MW, 188

*d.*  
Mononitrile: cryst. M.p. 94–5°.  $[\alpha]^{20}_D + 11.5^\circ$  in toluene.

*l.*  
Mononitrile: cryst. from Et<sub>2</sub>O. M.p. 90°.  $[\alpha]^{20}_D - 10.5^\circ$  in toluene.

*dl.*  
Di-Et ester:  $\text{C}_{13}\text{H}_{24}\text{O}_4$ . MW, 244. B.p. 143°/42 mm.  $D^{25}_D$  0.9803.  $n^{25}_D$  1.4239.

Monoamide:  $\text{C}_9\text{H}_{17}\text{O}_3\text{N}$ . MW, 187. Prisms from H<sub>2</sub>O. M.p. 137° decomp. Sol. EtOH, Me<sub>2</sub>CO. Spar. sol. ligroin, cold H<sub>2</sub>O.

Mononitrile: 1-isopropyl-1-cyanovaleric acid, 1-propyl-1-cyanoisovaleric acid.  $\text{C}_9\text{H}_{15}\text{O}_2\text{N}$ . MW, 169. M.p. 40–8°. B.p. 168–9°/13 mm. Very spar. sol. H<sub>2</sub>O. Et ester:  $\text{C}_{11}\text{H}_{19}\text{O}_2\text{N}$ . MW, 197. B.p. 242–3°/749 mm., 113–14°/11–12 mm.  $D^{20}_D$  0.943.

Shonle, Moment, *J. Am. Chem. Soc.*, 1923, 45, 248.

Fischer, Flatau, *Ber.*, 1909, 42, 2984.

**1-Propyl-2-isopropylsuccinic Acid** (2-Methylheptane-3:4-dicarboxylic acid)

$\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}(\text{COOH})_2$   
 $(\text{CH}_3)_2\text{CH}\cdot\text{CH}(\text{COOH})_2$   
 $\text{C}_{10}\text{H}_{18}\text{O}_4$  MW, 202

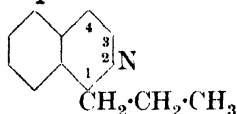
*Cis*:  
Plates from  $\text{CHCl}_3$ -pet. ether. M.p. 151–2°.  $k = 2.95 \times 10^{-4}$  at 25°. HCl at 200° → part. to *trans* form.

Anhydride:  $\text{C}_{10}\text{H}_{16}\text{O}_3$ . MW, 184. B.p. 265–75°/742 mm.

*Trans*:  
Cryst. from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 192–4°.  $k = 1.47 \times 10^{-4}$  at 25°. HCl at 200° → part. to *cis* form.

Bone, Sprankling, *J. Chem. Soc.*, 1900, 77, 660.

## 1-Propylisoquinoline



$C_{12}H_{13}N$  MW, 171

B.p. 140–60°/10 mm.

Picrate: m.p. 200–1°.

Späth, Berger, Kuntara, *Ber.*, 1930, **63**, 138.

## 3-Propylisoquinoline.

Oil with characteristic odour. B.p. 271°.  
 $D_4^{24}$  1.0156.

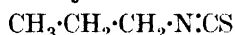
$B_2HAuCl_4$ : yellow needles. \*M.p. 118° decomp.

$B_2H_2PtCl_6$ : yellow needles. Decomp. at 189° without melting.

Picrate: m.p. 161°.

Albahary, *Ber.*, 1896, **29**, 2397.

## Propyl isothiocyanate



$C_4H_7NS$  MW, 101

B.p. 152.7°/743 mm.  $D_4^{16}$  0.9781.  $n_D^{16}$  1.5085.

Delépine, *Ann. chim. phys.*, 1912, **25**, 560.

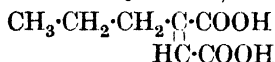
## 1-Propylisovaleric Acid.

See 2-Methylhexane-3-carboxylic Acid.

## 2-Propylkairoline.

See under 2-Propyl-1 : 2 : 3 : 4-tetrahydroquinoline.

**Propylmaleic Acid** (*Ethylcitraconic acid*, 1-pentene-1 : 2-dicarboxylic acid)



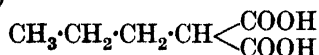
$C_7H_{10}O_4$  MW, 158

Prisms from  $H_2O$ , needles from  $Et_2O$ -ligroin. M.p. 93–5° (92–4°). Sol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. ligroin. Heat  $\rightarrow$  propylmaleic anhydride.  $H_2O$  at 135–50°  $\rightarrow$  mainly propylidene-succinic acid. NaHg  $\rightarrow$  propylsuccinic acid. Ag salt spar. sol.  $H_2O$ . Ba salt less sol. hot  $H_2O$  than cold.

Anhydride:  $C_7H_8O_3$ . MW, 140. Oil. B.p. 152–3°/68 mm.

Fittig, Glaser, *Ann.*, 1899, **304**, 184.

**Propylmalonic Acid** (*Butane-1 : 1-dicarboxylic acid*)



$C_6H_{10}O_4$  MW, 146

Plates from  $C_6H_6$ . M.p. 96°. Sol. to 45.6 parts in 100 parts  $H_2O$  at 0°.  $k = 1.13 \times 10^{-3}$  at 25°. At 180°  $\rightarrow$  *n*-valeric acid.

*Di-Me ester*:  $C_8H_{14}O_4$ . MW, 174. B.p. 203°/756 mm.  $D_4^{20}$  1.0398.  $n_D^{20}$  1.42155.

*Di-Et ester*:  $C_{10}H_{18}O_4$ . MW, 202. B.p. 221° (222–7°/750 mm.), 193.5–194.5°/330 mm.  $D_4^{15}$  0.99309,  $D_4^{25}$  0.98541.

*Diamide*:  $C_6H_{12}O_2N_2$ . MW, 144. Cryst. from  $H_2O$ . M.p. 184°. Spar. sol. EtOH, cold  $H_2O$ . Insol.  $Et_2O$ ,  $CHCl_3$ .

*Mononitrile*: 1-cyano-*n*-valeric acid, propylcyanoacetic acid.  $C_6H_9O_2N$ . MW, 127. Oil. B.p. 125–30°/0.2 mm. *Et ester*:  $C_8H_{13}O_2N$ . MW, 155. B.p. 218–19°/755 mm., 105–10°/15 mm.  $D_4^{32}$  0.972. *Amide*: 1-cyano-*n*-valeric acid, propyleyanacetamide.  $C_6H_{10}ON_2$ . MW, 126. Prisms from  $Et_2O$ . M.p. 124–124.5° (118°). B.p. 281°. Sol.  $Et_2O$ ,  $CHCl_3$ . Mod. sol. hot  $H_2O$ . *Anilide*: m.p. 88–9°.

*Dinitrile*:  $C_6H_8N_2$ . MW, 108. Oil. B.p. 210°/750 mm.  $D_4^{18}$  0.9224.

*Monohydrazide*:  $C_6H_{12}O_3N_2$ . MW, 160. Cryst. from  $H_2O$ . M.p. 139° decomp. *K salt*: m.p. 120°. Very hygroscopic.

*Dihydrazide*:  $C_6H_{14}O_2N_4$ . MW, 174. Prisms from EtOH. M.p. 158°. *B,2HCl*: m.p. 180°. *N : N'-Diacetyl*: m.p. 245°.

*Dianilide*: prisms. M.p. 198°.

*Di-p-toluidide*: prisms. M.p. 186°.

Fürth, *Monatsh.*, 1888, **9**, 309.

Bischoff, *Ber.*, 1895, **28**, 2619.

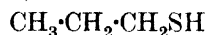
Fischer, Brieger, *Ber.*, 1915, **48**, 1520.

Henry, *Jahresber. Fortschr. Chem.*, 1889, 638.

Vogel, *J. Chem. Soc.*, 1934, 333.

Curtius, Lehmann, *J. prakt. Chem.*, 1930, 125, 224.

**Propyl Mercaptan** (1-Mercaptopropane, thiopropyl alcohol)



$C_3H_8S$  MW, 76

M.p. – 113.3°. B.p. 67°.  $D_4^0$  0.86169,  $D_4^{25}$  0.83572.  $n_D^{25}$  1.4351.

3 : 5-Dinitrobenzoyl: cryst. from AcOH.Aq. M.p. 51–2°.

Acid 3-nitrophthalate: cryst. from AcOH.Aq. M.p. 136–7°.

Hg(*S*- $C_3H_7$ )<sub>2</sub>: leaflets. M.p. 71–2°.

Ellis, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 1674.

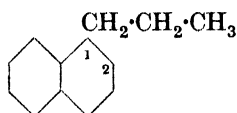
Wertheim, *J. Am. Chem. Soc.*, 1929, **51**, 3661.

**Propyl *p*-methoxyphenyl Ketone.**

See under *p*-Hydroxybutyrophenone.

**Propyl *p*-methoxyphenyl sulphide.**

See under Thiohydroquinone.

**1-Propylnaphthalene** $\text{C}_{13}\text{H}_{14}$ 

MW, 170

B.p. 274–5°.

Picrate : yellow needles. M.p. 140–1°.

Bargellini, Melacini, *Gazz. chim. ital.*, 1908, **38**, ii, 570.**2-Propylnaphthalene.**

B.p. 277–9°.

Picrate : yellow needles. M.p. 89–90°.

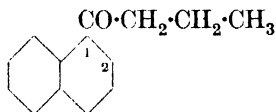
See previous reference.

*Note.*—Roblin, Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 158, claim that the data for the above two compounds should be interchanged.

**Propyl naphthyl Ether.**

See under Naphthol.

**Propyl 1-naphthyl Ketone** ( $\alpha$ -Butyronaphth-one)

 $\text{C}_{14}\text{H}_{14}\text{O}$ 

MW, 198

Yellow oil. B.p. 316–18°.  $D_4^{20}$  1.0861.  $n_D^{27}$  1.596. Very sol. most org. solvents.

Oxime : liq. B.p. 206–8°/13 mm.

Rousset, *Bull. soc. chim.*, 1896, **15**, 65.

**Propyl 2-naphthyl Ketone** ( $\beta$ -Butyronaphthone).

Prisms. M.p. 52°. B.p. 322–4°, 184–5°/16 mm.

Oxime : needles. M.p. 89°.

Azine : yellow needles. M.p. 130°.

Picrate : needles. M.p. 68–9°.

Comp. with  $\text{AlCl}_3$  : dark green needles. M.p. 92–5°.Barbot, *Bull. soc. chim.*, 1930, **47**, 1314.Perrier, *Bull. soc. chim.*, 1896, **15**, 322.Rousset, *Bull. soc. chim.*, 1896, **15**, 66.

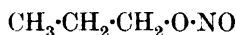
**Propylnitramine** (N-Nitropropylamine, 1-nitraminopropane)

 $\text{C}_3\text{H}_8\text{O}_2\text{N}_2$ 

MW, 104

Colourless liq. Cryst. on cooling. M.p. –21°. B.p. 128–9°/40 mm.  $D^{15}$  1.1046.Misc. with EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O → acid sol.Umbgrove, Franchimont, *Rec. trav. chim.*, 1898, **17**, 272.**Propyl nitrate** $\text{C}_3\text{H}_7\text{O}_3\text{N}$ 

MW, 105

B.p. 110.5°.  $D_4^{20}$  1.0548.  $n_D^{20}$  1.3979.Cowley, Partington, *J. Chem. Soc.*, 1933, 1252.Wallach, Schulze, *Ber.*, 1881, **14**, 421.**Propyl nitrite** $\text{C}_3\text{H}_7\text{O}_2\text{N}$ 

MW, 89

B.p. 46–8° (57°).  $D_4^{20}$  0.8861.  $n_D^{20}$  1.3604. H (+ Ni) at 130° → propylamine; at 200° → a mixture of mono-, di- and tri-propylamines.Cowley, Partington, *J. Chem. Soc.*, 1933, 1253.

**Propyl pentadecyl Ketone** (Nonadecanone-4)

 $\text{C}_{19}\text{H}_{38}\text{O}$ 

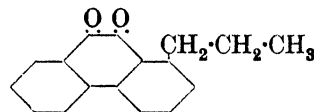
MW, 282

M.p. 50.5°. B.p. 211°/11 mm., part. decomp. Spar. sol. EtOH.

Oxime : exists in two forms. (i) M.p. 25.5–26.5°. (ii) M.p. 43.5–44.5°.

Bertrand, *Bull. soc. chim.*, 1896, **15**, 766.Furukawa, *Chem. Abstracts*, 1933, **27**, 2131.**2-Propyl-2-pentene.**

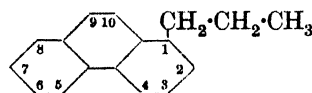
See 4-Methyl-3-heptene.

**1-Propylphenanthraquinone** $\text{C}_{17}\text{H}_{14}\text{O}_2$ 

MW, 250

Orange plates from EtOH. M.p. 139–40°.

Quinoxaline deriv. : pale yellow needles from AcOH. M.p. 144–5°.

Haworth, Mavin, Sheldrick, *J. Chem. Soc.*, 1934, 460.**1-Propylphenanthrene** $\text{C}_{17}\text{H}_{16}$ 

MW, 220

Plates from MeOH. M.p. 34–5°.

*Picrate*: yellow needles from MeOH. M 100–1°.

See previous reference.

### 9-Propylphenanthrene.

Plates from EtOH. M.p. 74°. B.p. 265–70°/22 mm.

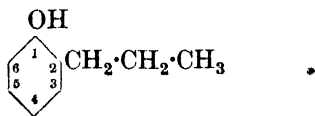
*Picrate*: yellow needles from EtOH. M.p. 134°.

Miller, Bachman, *J. Am. Chem. Soc.*, 1935, **57**, 768.

### Propylphenetole.

See under Propylphenol.

### *o*-Propylphenol (2-Hydroxy-1-propylbenzene)



$\text{C}_9\text{H}_{12}\text{O}$

MW, 136

Oil. B.p. 220–220.5°.  $D_{20}^{15}$  1.000.

*Me ether*: 2-methoxy-1-propylbenzene, *o*-propylanisole.  $\text{C}_{10}\text{H}_{14}\text{O}$ . MW, 150. B.p. 207–9°/757.7 mm.  $D^{20}$  0.96944.

*Et ether*: 2-ethoxy-1-propylbenzene, *o*-propylphenetole. B.p. 213°/754 mm., 99–100°/16 mm.  $D_{20}^{25}$  0.92396.  $n_D^{25}$  1.494.

*Propionyl*: b.p. 245°.

*Phenylurethane*: needles from formic acid. M.p. 111°.

Claissen, *Ann.*, 1919, **418**, 87.

Farinholt, Harden, Twiss, *J. Am. Chem. Soc.*, 1933, **55**, 3386.

### *m*-Propylphenol (3-Hydroxy-1-propylbenzene).

B.p. 228°. Spar. sol.  $\text{H}_2\text{O}$ .  $\text{FeCl}_3 \rightarrow$  green col. in EtOH, bluish col. in  $\text{H}_2\text{O}$ .

*Me ether*: 3-methoxy-1-propylbenzene, *m*-propylanisole. B.p. 212–13°.

*Et ether*: 3-ethoxy-1-propylbenzene, *m*-propylphenetole. B.p. 220–4°/753 mm., 109–10°/15 mm.  $D_{20}^{20}$  0.94558.  $n_D^{20}$  1.5025.

Henrard, *Chem. Zentr.*, 1907, II, 1512.

Ciamician, Silber, *Ber.*, 1890, **23**, 1162.

### *p*-Propylphenol (4-Hydroxy-1-propylbenzene).

Cryst. M.p. 21–2°. B.p. 230–2°, 120°/19 mm.  $D_4^{20}$  1.089.

*Me ether*: 4-methoxy-1-propylbenzene, *p*-propylanisole, dihydroanethole. B.p. 215–16°, 86.5°/10 mm.  $D_4^{20}$  0.94718.  $n_D^{20}$  1.5045.

*Et ether*: 4-ethoxy-1-propylbenzene, *p*-propylphenetole. B.p. 223–30°, 108–10°/13 mm.  $D_4^{15}$  0.94.

*Acetyl*: b.p. 245–6°/745 mm.  $D^{20}$  1.02904.

*Propionyl*: b.p. 254–6°.

*Benzoyl*: m.p. 37–8°.

*Salicyloyl*: m.p. 57°.

*Phenylurethane*: cryst. from EtOH. M.p. 128.5–129°.

Farinholt, Harden, Twiss, *J. Am. Chem. Soc.*, 1933, **55**, 3386.

Baranger, *Bull. soc. chim.*, 1931, **49**, 1213.

Albright, *J. Am. Chem. Soc.*, 1914, **36**, 2197.

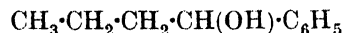
Clemmensen, *Ber.*, 1914, **47**, 53.

Ipatjew, *Ber.*, 1913, **46**, 3590.

### 1-Propyl-1-phenylbutylene-1.

See 4-Phenyl-3-heptene.

**Propylphenylcarbinol** (1-Phenyl-*n*-butyl alcohol,  $\alpha$ -hydroxybutylbenzene,  $\alpha$ -propylbenzyl alcohol)



$\text{C}_{10}\text{H}_{14}\text{O}$

MW, 150

*d*-.

M.p. 49°. B.p. 115°/14 mm.  $[\alpha]_{5461} + 52.2^\circ$  in  $\text{C}_6\text{H}_6$ .

*Acetyl*: b.p. 125°/16 mm.  $n_D^{20}$  1.4889.

*Acid phthalate*: m.p. 53–4°.  $[\alpha]_{5461} + 11.5^\circ$  in  $\text{Et}_2\text{O}$ .

*l*-.

M.p. 48–9°.  $[\alpha]_{5461} - 53.5^\circ$  in  $\text{C}_6\text{H}_6$ .

*Acid phthalate*: m.p. 52–3°.  $[\alpha]_{5461} - 11.0^\circ$  in  $\text{Et}_2\text{O}$ .

*dl*-.

Oil with aromatic odour. B.p. 168–70°/100 mm., 113–15°/10 mm.  $D_4^{13.7}$  0.9861.  $n_D^{13.7}$  1.51914.

*Acetyl*: b.p. 117–18°/8 mm.

*Acid phthalate*: m.p. 90–1°.

Grignard, *Ann. chim. phys.*, 1901, **24**, 466.

Kenyon, Partridge, *J. Chem. Soc.*, 1936, 128.

Klages, *Ber.*, 1904, **37**, 2312.

### Propyl-phenylethyl-carbinol.

See 1-Phenylhexanol-3 and 2-Phenylhexanol-3.

### Propyl phenylethyl Ketone.

See 1-Phenylhexanone-3 and 2-Phenylhexanone-3.

### Propylphenylglycollic Acid.

See 1-Hydroxy-1-phenylbutyric Acid.

### 3-Propyl-3-phenylhexane.

See 4-Éthyl-4-phenylheptane.

### Propyl phenyl Ketone.

See Butyrophenone.

### Propylphenylpropylene.

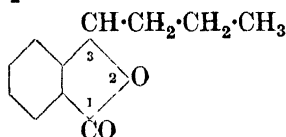
See 1-Phenylhexene-2.

**Propyl phenyl sulphone**

$\text{C}_6\text{H}_5\cdot\text{SO}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_3$   
 $\text{C}_9\text{H}_{12}\text{O}_2\text{S}$  MW, 184

Plates from ligroin. M.p.  $46^\circ$  ( $44^\circ$ ). Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. boiling H<sub>2</sub>O.

Baldwin, Robinson, *J. Chem. Soc.*, 1932, 1448.

**3-Propylphthalide**

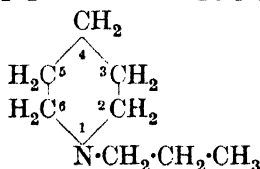
$\text{C}_{11}\text{H}_{12}\text{O}_2$  MW, 176

Oil with odour of celery. M.p.  $20^\circ$ . B.p.  $293-7^\circ/735$  mm. decomp.,  $243-7^\circ/220$  mm.,  $150^\circ/14$  mm.  $D_{15}^{15}$  1.1073.  $n_D^{17.5}$  1.5327. Volatile in steam. NaOH fusion  $\rightarrow$  butyric and benzoic acids.

Tasman, *Rec. trav. chim.*, 1927, 46, 653.

**N-Propylphthalimide.**

See under Phthalimide.

**N-Propylpiperidine (Propylpiperidylamine)**

$\text{C}_8\text{H}_{17}\text{N}$  MW, 127

B.p.  $149-50^\circ$ .

*B.HCl*: m.p.  $212-13^\circ$ . Hygroscopic.

*Chloroplatinate*: m.p.  $179^\circ$ .

*Picrate*: yellow needles. M.p.  $121^\circ$  ( $108^\circ$ ).

*Methiodide*: m.p.  $181-2^\circ$ .

*Ethiodide*: m.p.  $276.5^\circ$ .

v. Braun, *Ber.*, 1909, 42, 2048.

Auerbach, Wolfenstein, *Ber.*, 1899, 32, 2511.

Ladenburg, *Ber.*, 1881, 14, 1348.

**2-Propylpiperidine.**

See Coniine.

**3-Propylpiperidine.**

*dl.*

$[\alpha]_D^{15} + 5.9^\circ$ .

*B.HCl*: cryst. from Me<sub>2</sub>CO. M.p.  $147^\circ$ . Very sol. H<sub>2</sub>O, EtOH.

*l-Tartrate*: cryst. from H<sub>2</sub>O. M.p.  $161^\circ$ .

*l.*

B.p.  $174^\circ/752.5$  mm.  $D_4^{19}$  0.8517.  $[\alpha]_D^{19} - 6.6^\circ$ .

*B.HCl*: needles from Me<sub>2</sub>CO. M.p.  $147^\circ$ . Very sol. H<sub>2</sub>O, EtOH.

*d-Tartrate*: cryst. from H<sub>2</sub>O. M.p.  $161^\circ$ .

*dl.*

Liq. with odour resembling coniine. B.p.  $174^\circ/758$  mm.  $D_4^{25}$  0.8475. Sol. in about 80 parts H<sub>2</sub>O at ord. temp. Turns brown in air.

*B.HCl*: prisms or needles from H<sub>2</sub>O. M.p.  $127-9^\circ$ . Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

*B.HAuCl<sub>4</sub>*: lemon-yellow needles from H<sub>2</sub>O. M.p.  $95-8^\circ$ .

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-yellow needles. M.p.  $94^\circ$  (slow heat.),  $134^\circ$  (rapid heat.).

*Picrate*: yellow needles. M.p.  $121.5^\circ$ .

Granger, *Ber.*, 1895, 28, 1203; 1897, 30, 1060.

**4-Propylpiperidine.**

Liq. with odour resembling coniine. B.p.  $178-80^\circ$ .

Ahrens, *Ber.*, 1905, 38, 159.

**Propylpiperidylamine.**

See *N*-Propylpiperidine.

**Propylpropenylcarbinol.**

See 2-Heptenol-4.

**Propylpropenylethylene.**

See 2:4-Octadiene.

**Propyl propenyl Ketone.**

See 2-Heptenone-4.

**Propylpropionic Acid (1-Pentine-1-carboxylic acid)**

$\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{C}(\text{C}(\text{COOH}))$   
 $\text{C}_6\text{H}_8\text{O}_2$  MW, 112

Feathery cryst. M.p.  $27^\circ$ . B.p.  $126-7^\circ/24$  mm.,  $119-21^\circ/16$  mm. Sol. EtOH, Et<sub>2</sub>O, ligroin. Somewhat difficultly sol. H<sub>2</sub>O. Dist. at atmospheric press.  $\rightarrow$  propylacetylene. Hot KOH.Aq.  $\rightarrow$  methyl propyl ketone. Hot alc. KOH  $\rightarrow$  butyrylacetic acid.

*Me ester*:  $\text{C}_7\text{H}_{10}\text{O}_2$ . MW, 126. B.p.  $80-2^\circ/23$  mm.  $D_4^0$  0.9648.

*Et ester*:  $\text{C}_8\text{H}_{12}\text{O}_2$ . MW, 140. B.p.  $93-4^\circ/24$  mm.  $D_4^0$  0.9468.

*Isoamyl ester*:  $\text{C}_{11}\text{H}_{18}\text{O}_2$ . MW, 182. B.p.  $127-8^\circ/22$  mm.  $D_4^0$  0.9207.

Moureu, Delange, *Bull. soc. chim.*, 1903, 29, 652.

**Propylpropionylcarbinol.**

See 4-Heptanolone-3.

**2-Propylpropylene.**

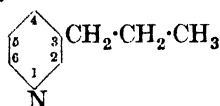
See 2-Methyl-1-pentene.

**2-Propylpyridine.**

See Conyryne.



## 3-Propylpyridine

 $C_8H_{11}N$ 

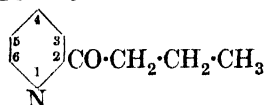
MW, 121

B.p. 170°. Ox.  $\rightarrow$  nicotinic acid.Cahours, Étard, *Compt. rend.*, 1881, 92, 1082.

## 4-Propylpyridine.

B.p. 184-6°.  $D_4^{20}$  0.9381. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.*B.HCl*: needles from EtOH.Aq. M.p. 215°.*B.HAuCl<sub>4</sub>*: yellow cryst. M.p. 113-15°.*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: brownish-yellow cryst. M.p. 204°.*Picrate*: needles from EtOH. M.p. 153°.Koenigs, Jaeschke, *Ber.*, 1921, 54, 1355.

## Propyl 2-pyridyl Ketone (2-Butyrylpyridine)

 $C_9H_{11}ON$ 

MW, 149

Oil with characteristic odour. B.p. 217-18° (216-20°). Sol. acids.

*Oxime*: needles from pet. ether. M.p. 48°.*Benzoyl*: yellow leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 56-7°.*Phenylhydrazone*: yellowish needles from EtOH. M.p. 82°. Unstable.*p-Sulphophenylhydrazone*: yellow needles. M.p. 251°.*Picrate*: yellow needles from H<sub>2</sub>O. M.p. 75°.*Methiodide*: yellow needles from EtOH-Et<sub>2</sub>O. M.p. 79°.Engler, Majmon, *Ber.*, 1891, 24, 2536.Pinner, *Ber.*, 1901, 34, 4243.

## Propyl 3-pyridyl Ketone (3-Butyrylpyridine).

Liq. with odour resembling coniine. B.p. 246-52°. Sol. EtOH, Et<sub>2</sub>O, acids.*Phenylhydrazone*: yellow cryst. M.p. 182° (129-30°).*Semicarbazone*: m.p. 169-70°.*Ethiodide*: yellow cryst. M.p. 192°.La Forge, *J. Am. Chem. Soc.*, 1928, 50, 2477.Engler, *Ber.*, 1891, 24, 2541.

## Propyl 4-pyridyl Ketone (4-Butyrylpyridine).

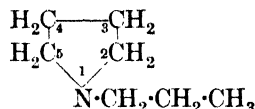
B.p. 229-31°.

*Picrate*: yellow needles from H<sub>2</sub>O. M.p. 96°.Pinner, *Ber.*, 1901, 34, 4252.

## Propylpyrogallol dimethyl Ether.

See Picamar.

## N-Propylpyrrolidine

 $C_7H_{15}N$ 

MW, 113

B.p. 130°.  $D_4^{20}$  0.8171.  $n_D^{20}$  1.4389. Sol. H<sub>2</sub>O.*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: red cryst. Decomp. at 184-90°.*Picrate*: yellow plates. M.p. 105° (101°).v. Braun, *Ber.*, 1911, 44, 1254.Jurjew, Schenjan, *Chem. Zentr.*, 1936, I, 4293.

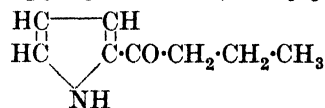
## 2-Propylpyrrolidine.

Oil with odour resembling piperidine. B.p. 145-50°/765 mm. Sol. H<sub>2</sub>O with alk. reaction.*Chloroaurate*: yellow needles or leaflets. M.p. 120°.*Chloroplatinate*: m.p. anhyd. 135°.*Picrate*: m.p. 104-104.5°.*N-Me*: C<sub>8</sub>H<sub>17</sub>N. MW, 127. B.p. 146-7°.  $D_4^{20}$  0.815. *B.HAuCl<sub>4</sub>*: yellow leaflets. M.p. 76°.*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange leaflets. M.p. 145-6°.*Picrate*: yellow needles from EtOH. M.p. 124°.*N-Benzenesulphonyl*: needles from 80% EtOH. M.p. 66-67.5°.Hess, *Ber.*, 1913, 46, 4110.Löffler, *Ber.*, 1910, 43, 2039.Gabriel, *Ber.*, 1909, 42, 1264.

## 3-Propylpyrrolidine.

Oil with odour resembling piperidine. B.p. 158-60°/746 mm.  $D_4^{20}$  0.8450.  $n_D^{20}$  1.4469. Fumes in air. Absorbs H<sub>2</sub>O and CO<sub>2</sub> from the air.Longinow, *Chem. Zentr.*, 1915, I, 982.

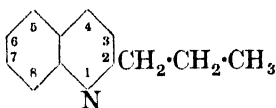
## Propyl 2-pyrryl Ketone (2-Butyrylpyrrole)

 $C_8H_{11}ON$ 

MW, 137

Cryst. from H<sub>2</sub>O. M.p. 48.5°. B.p. 235°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, pet. ether, KOH.Aq.*Phenylhydrazone*: yellow cryst. from pet. ether. M.p. 80.5°.*Semicarbazone*: needles from H<sub>2</sub>O. M.p. 131°.Tschelinzeff, Terentjeff, *Ber.*, 1914, 47, 2650.Oddo, *Ber.*, 1910, 43, 1016.

## 2-Propylquinoline

 $C_{12}H_{13}N$ 

MW, 171

Pale yellowish-green oil. B.p.  $142-5^{\circ}/13$  mm.,  $130-1^{\circ}/10$  mm.  $D_4^{25}$  1.038.  $n_D^{25}$  1.5886.

*Chloromercurate* : m.p.  $112^{\circ}$ .

*Methiodide* : m.p.  $184^{\circ}$ .

*Picrate* : yellow needles or leaflets from EtOH. M.p.  $163-4^{\circ}$ .

*Methopicate* : yellow needles. M.p.  $118^{\circ}$ .

Freund, Kessler, *J. prakt. Chem.*, 1918, **98**, 233.

Meisenheimer, Schütze, *Ber.*, 1923, **56**, 1353.

Delaby, Hiron, *Compt. rend.*, 1930, **191**, 845.

## 4-Propylquinoline.

Liq. with odour resembling quinoline. B.p.  $159^{\circ}/16$  mm.

*Hydrochloride* : m.p.  $156-7^{\circ}$ .

$B, HBr, CdBr_2$  : cryst. from EtOH. M.p.  $148^{\circ}$ .

$B_2, 2HCl, HgCl_2$  : cryst. from EtOH. M.p.  $148^{\circ}$ .

$B_2, H_2PtCl_6$  : cryst. from conc. HCl. M.p.  $198^{\circ}$  decomp.

*Picrate* : yellow needles from EtOH. M.p.  $204^{\circ}$ .

Koenigs, *Ber.*, 1898, **31**, 2376.

Blaise, Maire, *Bull. soc. chim.*, 1908, **3**, 667.

## 8-Propylquinoline.

Pale yellow liq. B.p.  $142^{\circ}/15$  mm.

$B_2, H_2PtCl_6$  : m.p.  $196^{\circ}$ . Very spar. sol. hot  $H_2O$ .

*Picrate* : yellowish-red needles from EtOH. M.p.  $142^{\circ}$ .

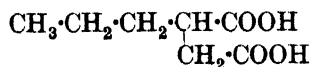
*Methiodide* : m.p.  $136^{\circ}$ .

v. Braun, Heider, Wyczatkowska, *Ber.*, 1918, **51**, 1215.

## Propylresorcinol.

See 2 : 4-Dihydroxy-1-propylbenzene.

**Propylsuccinic Acid** (*Pentane-1 : 2-dicarboxylic acid*)

 $C_7H_{12}O_4$ 

MW, 160

*d.*

M.p.  $93.9^{\circ}$ .  $[\alpha]_D + 9.6^{\circ}$ .

*dl.*

Cryst. from  $H_2O$  or  $C_6H_6$ . M.p.  $100.5^{\circ}$  ( $92-3^{\circ}$ ). 2.83 parts sol. 100 parts cold  $CHCl_3$ .  $k$  (first) =  $8.9 \times 10^{-5}$  at  $25^{\circ}$ ; (second) =  $1.2 \times 10^{-6}$  at  $100^{\circ}$ .

*Di-Me ester* :  $C_9H_{16}O_4$ . MW, 188. B.p.  $112^{\circ}/15$  mm.,  $107^{\circ}/11$  mm.

*Di-Et ester* :  $C_{11}H_{20}O_4$ . MW, 216. B.p.  $132-4^{\circ}/25$  mm.

*Diamide* :  $C_7H_{14}O_2N_2$ . MW, 158. Needles from EtOH. M.p.  $234-5^{\circ}$ . Spar. sol. EtOH.

*Anhydride* :  $C_7H_{10}O_3$ . MW, 142. Viscous liq. B.p.  $145-55^{\circ}/20$  mm.

*Dihydrazide* : powder from EtOH. M.p.  $176^{\circ}$ .

Locquin, *Bull. soc. chim.*, 1909, **5**, 1073.

Fittig, Glaser, *Ann.*, 1899, **304**, 188.

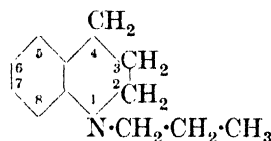
Timmermans, van der Haegen, *Bull. soc. chim. Belg.*, 1933, **42**, 448.

Scheibler, Schmidt, *Ber.*, 1921, **54**, 153.

## 2-Propyl-1 : 4 : 5 : 6-tetrahydropyridine.

See  $\gamma$ -Coniceine.

## N-Propyl-1 : 2 : 3 : 4-tetrahydroquinoline

 $C_{12}H_{17}N$ 

MW, 175

Colourless liq. B.p.  $146^{\circ}/16$  mm. Gradually turns reddish-brown.

$B, HCl$  : m.p.  $162^{\circ}$ .

$B, HBr$  : m.p.  $177^{\circ}$ . Somewhat difficultly sol. cold  $H_2O$ .

$B, HI$  : needles. M.p.  $178^{\circ}$ . Insol. cold  $H_2O$ .

*Picrate* : m.p.  $73^{\circ}$ .

*Methiodide* : kairolin propiodide. Plates from EtOH-Et<sub>2</sub>O. M.p.  $135^{\circ}$ .

v. Braun, *Ber.*, 1909, **42**, 2222.

## 2-Propyl-1 : 2 : 3 : 4-tetrahydroquinoline.

Oil with faint violet fluor. B.p.  $258^{\circ}/746$  mm.,  $152^{\circ}/20$  mm.,  $140-1^{\circ}/10$  mm.  $D_4^{25}$  0.959.  $n_D^{25}$  1.5673.

$B, HCl$  : m.p.  $221^{\circ}$ . Spar. sol. EtOH, hot  $H_2O$ . Sublimes.

*Picrate* : orange plates from EtOH or Et<sub>2</sub>O. M.p.  $125^{\circ}$ .

*N-Me* : 2-propylkairolin.  $C_{13}H_{19}N$ . MW, 189. B.p.  $272-6^{\circ}$ ,  $151-7^{\circ}/20$  mm.,  $144.5^{\circ}/10$  mm. Volatile in steam. *Picrate* : yellow leaflets from EtOH. M.p.  $123^{\circ}$  ( $120^{\circ}$ ). *Methiodide* : melts between  $180-200^{\circ} \rightarrow$  its components.

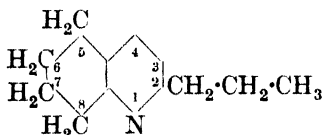
*N*-Benzoyl: leaflets from EtOH or Et<sub>2</sub>O. M.p. 102° (97°).

v. Braun, Gmelin, Petzold, *Ber.*, 1924, 57, 382.

Tröger, Ungar, *J. prakt. Chem.*, 1926, 112, 254.

Meisenheimer, Schütze, *Ber.*, 1923, 56, 1357.

### 2-Propyl-5:6:7:8-tetrahydroquinoline



C<sub>12</sub>H<sub>17</sub>N

MW, 175

B.p. 130-2°/11 mm.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: decomp. at 62°.

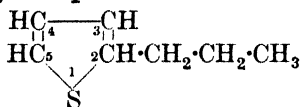
Picrate: yellow cryst. from EtOH. M.p. 119°.

See first reference above.

### 1-Propyltetramethylene Glycol.

See Heptandiol-1:4.

### 2-Propylthiophene



C<sub>7</sub>H<sub>10</sub>S

MW, 126

B.p. 157-60°. D<sub>20</sub><sup>20</sup> 0.9700, D<sub>4</sub><sup>20</sup> 0.9683. n<sub>D</sub><sup>20</sup> 1.5048.

Scheibler, Schmidt, *Ber.*, 1921, 54, 149.

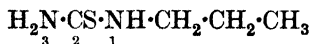
Steinkopf, Schubart, *Ann.*, 1921, 424, 21.

### 3-Propylthiophene.

B.p. 160-2°. D<sub>20</sub><sup>20</sup> 0.9733, D<sub>4</sub><sup>20</sup> 0.9716. n<sub>D</sub><sup>20</sup> 1.5057. Isatin + H<sub>2</sub>SO<sub>4</sub> → blue col.

Scheibler, Schmidt, *Ber.*, 1921, 54, 153.

### Propylthiourea



C<sub>4</sub>H<sub>10</sub>N<sub>2</sub>S

MW, 118

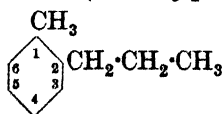
Needles from EtOH. M.p. 110°. Sol. EtOH. Mod. sol. H<sub>2</sub>O.

3-*N*-Me: plates from EtOH.Aq. M.p. 79°. Sol. EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>. Mod. sol. H<sub>2</sub>O. Insol. ligroin.

3-*N*-Et: plates from EtOH. M.p. 52°.

Hecht, *Ber.*, 1890, 23, 283.

### *o*-Propyltoluene (2-Methylpropylbenzene)



C<sub>10</sub>H<sub>14</sub>

MW, 134

B.p. 184°, 65-8°/14 mm. D<sub>4</sub><sup>19</sup> 0.8747. n<sub>D</sub><sup>20</sup> 1.4995.

Claus, Hansen, *Ber.*, 1880, 13, 897.

Auwers, *Ann.*, 1919, 419, 111.

Kuhn, Deutsch, *Ber.*, 1932, 65, 48.

### *m*-Propyltoluene (3-Methylpropylbenzene).

B.p. 181.5-182.5°. D<sub>4</sub><sup>17</sup> 0.8648, D<sub>4</sub><sup>20</sup> 0.862. n<sub>D</sub><sup>20</sup> 1.4951.

See second reference above and also

Claus, Stüsser, *Ber.*, 1880, 13, 899.

### *p*-Propyltoluene (4-Methylpropylbenzene).

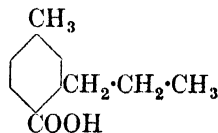
B.p. 183-4°. D<sub>4</sub><sup>15-4</sup> 0.8642. n<sub>D</sub><sup>20</sup> 1.4823.

Bayrac, *Bull. soc. chim.*, 1895, 13, 894.

Auwers, *Ann.*, 1919, 419, 112.

Ipat'ev, Orlov, Petrov, *Chem. Abstracts*, 1931, 25, 4540.

### 3-Propyl-*p*-toluic Acid (4-Methyl-2-propylbenzoic acid)



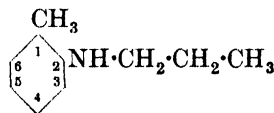
C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>

MW, 178

Needles from H<sub>2</sub>O. M.p. 75-6°. Volatile in steam.

Claus, *J. prakt. Chem.*, 1892, 46, 495.

### *N*-Propyl-*o*-toluidine



C<sub>10</sub>H<sub>15</sub>N

MW, 149

Oil. B.p. 230°.

Bischoff, Mintz, *Ber.*, 1892, 25, 2319.

### *N*-Propyl-*p*-toluidine.

Oil with odour of caraway. B.p. 235°/761 mm. D<sub>20</sub><sup>20</sup> 0.9243, D<sub>20</sub><sup>25</sup> 0.9172. n<sub>D</sub><sup>20</sup> 1.5367.

B, HCl: needles. M.p. 150-1°.

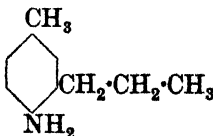
B<sub>2</sub>(COOH)<sub>2</sub>: cryst. M.p. 116-17°.

B<sub>2</sub>(COOH)<sub>2</sub>: m.p. 172-3° decomp.

Hori, Morley, *J. Chem. Soc.*, 1891, 59, 35.

Bischoff, Mintz, *Ber.*, 1892, 25, 2321.

### 3-Propyl-*p*-toluidine (4-Methyl-2-propylaniline)



C<sub>10</sub>H<sub>15</sub>N

MW, 149

B.p. 98–9°/13 mm.  $D_4^{25}$  0.9666.

$B, HCl$ : cryst. from  $H_2O$ . M.p. 195°.

$N$ -Benzoyl: m.p. 174.5°.

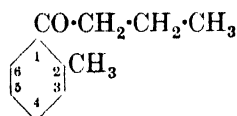
Picrate: cryst. from EtOH. M.p. 201°.

v. Braun, Bayer, Blessing, *Ber.*, 1924, 57, 402.

### Propyl tolyl Ether.

See under Cresol.

**Propyl o-tolyl Ketone** (2-Methylbutyrophenone)



$C_{11}H_{14}O$

MW, 162

B.p. 238.5°/758 mm.  $D_4^{20}$  0.9936.

Semicarbazone: m.p. 176°.

Senderens, *Bull. soc. chim.*, 1911, 9, 949.

**Propyl m-tolyl Ketone** (3-Methylbutyrophenone).

B.p. 247°/758 mm.  $D_4^{20}$  0.9882.

Semicarbazone: m.p. 152°.

See previous reference.

**Propyl p-tolyl Ketone** (4-Methylbutyrophenone).

B.p. 251.5°/758 mm. Sol. EtOH,  $Et_2O$ .

Semicarbazone: m.p. 190° (232°).

Phenylhydrazone: m.p. 73°.

See previous reference and also

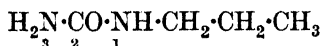
Willgerodt, Hambrecht, *J. prakt. Chem.*, 1910, 81, 78.

Blaise, *Compt. rend.*, 1901, 133, 1217.

### 1-Propyltricarballic Acid.

See Hexane-1 : 2 : 3-tricarboxylic Acid.

### Propylurea



$C_4H_{10}ON_2$  MW, 102

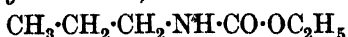
Prisms from EtOH. M.p. 110°. Sol. EtOH. Mod. sol.  $H_2O$ .

3-Acetyl: plates from  $CS_2$ . M.p. 115°. Mod. sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

Hecht, *Ber.*, 1890, 23, 283.

Mauguin, *Ann. chim. phys.*, 1911, 22, 343.

**Propylurethane** (Ethyl propylaminoformate, ethyl propylcarbamate)



$C_6H_{13}O_2N$  MW, 131

B.p. 191.5–192.5°/758 mm.

Schreiner, *J. prakt. Chem.*, 1880, 21, 125.

Nirdlinger, Acree, *Am. Chem. J.*, 1910, 43, 378.

### 1-Propylvaleric Acid.

Dipropylacetic Acid, *q.v.*

### 3-Propylvaleric Acid.

See 3-Methyl-n-heptylic Acid.

### Propylveratrol.

See under Cerylignol and under 2 : 3-Dihydroxy-1-propylbenzene.

### Propylvinylcarbinol.

See 1-Hexenol-3.

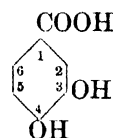
### Propyl vinyl Ketone.

See 1-Hexenone-3.

### Prothebenine.

See under Thebenine.

**Protocatechuic Acid** (3 : 4-Dihydroxybenzoic acid, catechol-4-carboxylic acid)



$C_7H_6O_4$

MW, 154

Needles +  $1H_2O$  from  $H_2O$ . M.p. 199°. Sol. EtOH. Mod. sol.  $H_2O$ . Insol.  $C_6H_6$ .  $k = 3.3 \times 10^{-5}$  at 25°. Aq. sol. with  $FeCl_3 \rightarrow$  green col.  $\rightarrow$  dark red with  $NaHCO_3$ .

3-Me ether: see Vanillic Acid.

4-Me ether: see Iovanillic Acid.

Di-Me ether: see Veratric Acid.

Di-Et ether: 3 : 4-diethoxybenzoic acid.

$C_{11}H_{14}O_4$ . MW, 210. Needles from EtOH.

M.p. 165–6°.  $k = 3.4 \times 10^{-5}$  at 25°. Et ester:

$C_{13}H_{18}O_4$ . MW, 238. Cryst. from EtOH. M.p. 56–7°.

Me ester:  $C_8H_8O_4$ . MW, 168. Needles from  $H_2O$ . M.p. 134.5°. Sol. EtOH. Spar. sol.  $H_2O$ . Has antimicrobial properties. 3-Benzoyl: m.p. 153.5–155°. 3-Acetyl-4-benzoyl: cryst. from MeOH. M.p. 102–3°. 4-Acetyl-3-benzoyl: prisms. M.p. 54–5°.

Et ester:  $C_9H_{10}O_4$ . MW, 182. Prisms from  $H_2O$ . M.p. 133–4°. Sol. EtOH.

Phenyl ester:  $C_{13}H_{10}O_4$ . MW, 230. Cryst. from EtOH. Aq. M.p. 189°.  $FeCl_3 \rightarrow$  green col.

Amide:  $C_7H_7O_3N$ . MW, 153. Cryst. from  $H_2O$ . M.p. 212°.

Nitrile:  $C_7H_5O_2N$ . MW, 135. Needles from  $H_2O$ . M.p. 156°. Sol.  $H_2O$ , EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ , ligroin, xylene. Diacetyl: needles from EtOH. M.p. 87°. Dibenzoyl: cryst. from EtOH. M.p. 131°.

Anilide: prisms from EtOH. M.p. 166–7°.

3-Acetyl: prisms from  $H_2O$ . M.p. 202–3°.

4-Benzoyl: cryst. from  $C_6H_6$ . M.p. 154–5°.

Diacetyl: cryst. from  $H_2O$ . M.p. 157–8°.

3-Benzoyl: microneedles. M.p. 225-7°.

Hoesch, Zarzecki, *Ber.*, 1917, **50**, 462.

Fischer, Bergmann, Lipschitz, *Ber.*, 1918, **51**, 45.

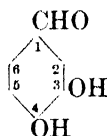
Schmidt, E.P., 145,081, (*Chem. Abstracts*, 1920, **14**, 3089); D.R.P., 278,778, (*Chem. Zentr.*, 1914, II, 1080).

Ono, Imoto, *J. Chem. Soc. Japan*, 1935, **56**, 715.

Pratt, Perkins, *J. Am. Chem. Soc.*, 1918, **40**, 224.

Miller, *Ann.*, 1883, **220**, 116.

**Protocatechuic Aldehyde** (3:4-Dihydroxybenzaldehyde)



$C_7H_6O_3$

MW, 138

Cryst. from toluene. M.p. 153°.

Oxime: m.p. 157° decomp.

Phenylhydrazone: two forms. (i) M.p. 175-6° decomp. (ii) M.p. 121-8°.

Semicarbazone: decomp. at 230°.

Azine: cryst. from EtOH.Aq. Decomp. about 245°.

2:4-Dinitrophenylhydrazone: dark red micro-cryst. from MeOH. M.p. 275° decomp.

3-Me ether: see Vanillin.

4-Me ether: see Isovanillin.

Di-Me ether: see Veratric Aldehyde.

3-Et ether:  $C_9H_{10}O_3$ . MW, 166. Plates from  $H_2O$ . M.p. 77-5°. 4-Benzyl ether: cryst. M.p. 57°.

Di-Et ether:  $C_{11}H_{14}O_3$ . MW, 194. Colourless oil. B.p. 278-80°.

3-Propyl ether:  $C_{10}H_{12}O_3$ . MW, 180. Needles from  $H_2O$ . M.p. 82°.

3-Isobutyl ether:  $C_{11}H_{14}O_3$ . MW, 194. Needles from EtOH.Aq. M.p. 94°.

3-Benzyl ether: m.p. 113-14°.

4-Benzyl ether: plates from EtOH. M.p. 122°.

Methylene ether: see Piperonal.

Ethylene ether:  $C_9H_8O_3$ . MW, 164. Needles from ligroin. M.p. 51-5°. B.p. 299°. Oxime: cryst. from EtOH. M.p. 75-75.5°.

Phenylhydrazone: cryst. from EtOH. M.p. 107-8°.

Azine: yellow needles from AcOH. M.p. 190-1°.

Carbonate: m.p. 124°. B.p. 289°, 162°/13 mm. Mod. sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ , AcOH. Spar. sol. Et<sub>2</sub>O,  $CCl_4$ .

3-Acetyl: plates from  $C_6H_6$ . M.p. 109-10°.

p-Nitrophenylhydrazone: red needles from EtOH. M.p. 195°. 4-Benzoyl: needles from EtOH. M.p. 109°.

Phenylhydrazone of 3-acetyl-4-benzoyl: needles from EtOH. M.p. 158°.

Diacetyl: cryst. from EtOH. M.p. 54°.

Phenylhydrazone: needles from EtOH. M.p. 135°. Semicarbazone: needles from EtOH. M.p. 200-2° decomp.

3-Benzoyl: needles from EtOH. M.p. 136-7°.

Phenylhydrazone: cryst. from EtOH. M.p. 192°.

4-Acetyl: cryst. from EtOH. M.p. 68°.

Phenylhydrazone of 4-acetyl-3-benzoyl: yellow plates from EtOH. M.p. 166°.

Dibenzoyl: phenylhydrazone, yellow plates. M.p. 167°.

Di-carbomethoxyl: m.p. 99-100°. p-Nitrophenylhydrazone: m.p. 187-9°.

Gattermann, *Ann.*, 1907, **357**, 374.

Boehringer, D.R.P., 269,544, (*Chem. Zentr.*, 1914, I, 591).

Schmidt, D.R.P., 295,337, (*Chem. Zentr.*, 1917, I, 41).

Hoesch, Zarzecki, *Ber.*, 1917, **50**, 465.

Tiemann, Koppe, *Ber.*, 1881, **14**, 2015.

Pacsu, v. Vargha, *Ber.*, 1926, **59**, 2818.

Fröschl, Bomberg, *Monatsh.*, 1927, **48**, 571.

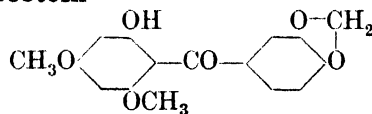
Roberts, E.P., 417,072, (*Chem. Abstracts*, 1935, **57**, 1099).

Vanillin Fabrik., D.R.P., 591,888, (*Chem. Zentr.*, 1934, I, 2491).

### Protocetraric Acid.

See Obtusatic Acid.

### Protocotoin



$C_{16}H_{14}O_6$

MW, 302

Occurs in Coto bark. Prisms from MeOH. M.p. 141-2°. Sol. Et<sub>2</sub>O,  $CHCl_3$ , AcOH,  $C_6H_6$ . Insol.  $H_2O$ . Sol. alkalis with yellow col. 65%  $HNO_3$  → bluish-green col. turning red on warming.  $FeCl_3$  in EtOH.Aq. → reddish-brown col.  $H_2SO_4$  → orange col.

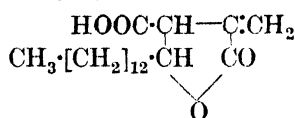
Me ether: oxyleukotin, methylprotocotoin.  $C_{17}H_{16}O_6$ . MW, 316. Prisms from EtOH. M.p. 134-5°. Sol. EtOH, AcOH. Mod. sol. Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ . 65%  $HNO_3$  → green col. turning reddish-brown on warming.  $H_2SO_4$  → orange col. No. col. with  $FeCl_3$  in EtOH.Aq. Phenylhydrazone: prisms from EtOH. M.p. 211°. Acetyl: cryst. from EtOH. M.p. 103°.

Späth, Bretschneider, *Monatsh.*, 1928, **49**, 429.

Houben, Fischer, *J. prakt. Chem.*, 1929, **123**, 89.

Ciamician, Silber, *Ber.*, 1893, **26**, 779; 1891, **24**, 2984.

## Protolichesteric Acid

 $\text{C}_{19}\text{H}_{32}\text{O}_4$ 

MW, 324

*l*-

Obtained from Japanese sub-alpine moss. Plates from AcOH. M.p. 107.5°.  $[\alpha]_D^{27}$  — 12.71°.

Semicarbazone: m.p. 140°.

Pyrazoline deriv.: plates from pet. ether. M.p. 54–5°.  $[\alpha]_D^{18}$  — 183.1°.

*d*-

Obtained from European Iceland moss. Plates from AcOH. M.p. 106°.  $[\alpha]_D^{20}$  + 12.07°.

Pyrazoline deriv.: plates. M.p. 54–5°.  $[\alpha]_D^{18}$  + 190.6°.

*l*- (Allo-).

Plates from AcOH. M.p. 88°.  $[\alpha]_D^{20}$  — 49.53°.

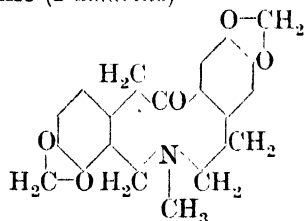
Pyrazoline deriv.: plates from pet. ether. M.p. 68–9°.  $[\alpha]_D^{18}$  — 73.69°.

Asahina, Asano, *J. Pharm. Soc. Japan*, 1927, **539**, 1.

Asano, Kanematsu, *Ber.*, 1932, **65**, 1175.

Asahina, Yanagita, *Ber.*, 1936, **69**, 120.

## Protopine (Fumarine)

 $\text{C}_{20}\text{H}_{19}\text{O}_5\text{N}$ 

MW, 353

Widely distributed in the *Papaveraceae* and *Fumariaceae*. Prisms from MeOH. M.p. 208°. Sol.  $\text{CHCl}_3$ . Spar. sol. MeOH, EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . AcOH sol.  $\longrightarrow$  deep bluish-violet col. with  $\text{H}_2\text{SO}_4$ .

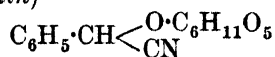
Methiodide: cryst. from MeOH. M.p. 217°.

Methosulphate: prisms from MeOH. M.p. 252°.

Perkin, *J. Chem. Soc.*, 1916, **109**, 1023.

Haworth, Perkin, *J. Chem. Soc.*, 1926, 1769.

**Prulaurasine** (dl-Mandelonitrile-d- $\beta$ -glucoside, laurocerolin)

 $\text{C}_{14}\text{H}_{17}\text{O}_6\text{N}$ 

MW, 295

Occurs in leaves of common cherry laurel (*Prunus laurocerasus*). Prisms or needles. M.p.

122–122.5°.  $[\alpha]_D$  — 54°. Sol.  $\text{H}_2\text{O}$ , MeOH. Insol.  $\text{Et}_2\text{O}$ . Hyd.  $\longrightarrow$  dl-mandelonitrile + glucose.

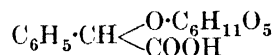
Tetra-acetyl: needles. M.p. 120–3°. Sol. EtOH,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ , AcOEt,  $\text{C}_6\text{H}_6$ . Insol. ligroin.

Hérissey, *Compt. rend.*, 1905, **41**, 959.

Caldwell, Courtauld, *J. Chem. Soc.*, 1907, **91**, 671.

Fischer, Bergmann, *Ber.*, 1917, **50**, 1062.

**Prulaurasinic Acid** (dl-Mandelic acid  $\beta$ -d-glucoside)

 $\text{C}_{14}\text{H}_{18}\text{O}_8$ 

MW, 314

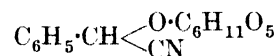
White hygroscopic powder. Cryst. with 1EtOH.  $[\alpha]_D^{11}$  — 28.17° to — 33.18°. Hyd. by emulsin. Does not reduce Fehling's.

Tetra-acetyl: needles. M.p. 130–50°.  $[\alpha]_D^{15}$  — 36.97° to — 43.46°. Et ester: needles from EtOH. M.p. 102–9°.  $[\alpha]_D$  — 33° to — 40.1° in  $\text{C}_6\text{H}_6$ . Sol. EtOH,  $\text{Me}_2\text{CO}$ , AcOEt,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ , pet. ether.

Karrer, Nägeli, Weidmann, *Helv. Chim. Acta*, 1919, **2**, 257.

Fischer, Bergmann, *Ber.*, 1917, **50**, 1053.

## Prunasine (d-Mandelonitrile-d-glucoside)

 $\text{C}_{14}\text{H}_{17}\text{O}_6\text{N}$ 

MW, 295

Occurs in *Prunus laurocerasus*, *P. padus* and *P. cerasus*. Needles from AcOEt. M.p. 147–50°.  $[\alpha]_D$  — 27°. Sol.  $\text{H}_2\text{O}$ , MeOH,  $\text{Me}_2\text{CO}$ . Alkalis  $\longrightarrow$  prulaurasine. HCl  $\longrightarrow$  d-mandelic acid.

Tetra-acetyl: needles from EtOH. M.p. 125–6°.  $[\alpha]_D^{22}$  — 52.5°.

Fischer, *Ber.*, 1895, **28**, 1508.

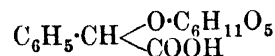
Caldwell, Courtauld, *J. Chem. Soc.*, 1907, **91**, 666, 671.

Auld, *J. Chem. Soc.*, 1908, **93**, 1276.

Fischer, Bergmann, *Ber.*, 1917, **50**, 1047.

Note.—All references state prunasine is derived from *l*-mandelonitrile, but see note under Mandelic Acid, Vol. II, p. 535, col. 2.

**Prunasinic Acid** (d-Mandelic acid- $\beta$ -d-glucoside)

 $\text{C}_{14}\text{H}_{18}\text{O}_8$ 

MW, 314

$[\alpha]_D^{15}$  — 138.6°. Sol.  $\text{H}_2\text{O}$ .



## Pterocarpine

$C_{17}H_{14}O_5$   $C_{16}H_{11}O_4(OCH_3)$  MW, 298

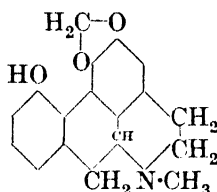
Obtained from red sandalwood. Needles from  $CCl_4$ . M.p.  $165^\circ$ . Insol.  $H_2O$ , cold EtOH,  $CS_2$ , acids, alkalis. Spar. sol. Et<sub>2</sub>O.  $[\alpha]_D^{20} - 220.1^\circ$ .

*Dinitrophenylhydrazones*: dark brown needles. M.p.  $305^\circ$  decomp.

Raudnitz, Perlmann, *Ber.*, 1935, 68, 1862.  
Leonhardt, Fay, *Arch. Pharm.*, 1935, 273, 53.

Dieterle, Leonhardt, *Arch. Pharm.*, 1929, 267, 81.

## Pukateine



$C_{18}H_{17}O_3N$  MW, 295

*l.*  
Obtained from the bark of the pukatea (*Laurelia Novae Zeelandiae*). Cryst. from Et<sub>2</sub>O. M.p.  $200^\circ$ . B.p.  $210-15^\circ/2$  mm. Sol. EtOH, Et<sub>2</sub>O,  $CHCl_3$ . Very sol. pyridine. Spar. sol. pet. ether. Insol.  $H_2O$ .  $[\alpha]_D^{15} - 220^\circ$  in EtOH.  $H_2SO_4 \rightarrow$  orange sol. changing to red and violet on warming.  $HNO_3 \rightarrow$  vermilion sol., orange on warming.  $K_2Cr_2O_7$  in  $H_2SO_4 \rightarrow$  purple col., excess of reagent  $\rightarrow$  green col. Has an action quantitatively similar to morphine on central nervous system.

*Acetyl*: methiodide, needles from  $Me_2CO$ . M.p.  $245^\circ$ .

*Me ether*: cryst. from dil. EtOH. M.p.  $137^\circ$ .  $[\alpha]_D - 261^\circ$ .  $H_2SO_4 \rightarrow$  orange to mauve col.  $HNO_3 \rightarrow$  brown sol. *B, HCl*: cryst. from EtOH. M.p.  $281^\circ$ . *B, HBr*: cryst. from EtOH. M.p.  $234^\circ$ . *d-Tartrate*: needles from EtOH.Aq. M.p.  $234^\circ$ .  $[\alpha]_D^{20} - 149.1^\circ$  in EtOH. *Methiodide*: cryst. M.p.  $240-1^\circ$ .

*d.*

*Me ether*: cryst. from EtOH.Aq. M.p.  $136^\circ$ .  $[\alpha]_D^{20} + 256.4^\circ$  in EtOH. *l-Tartrate*: cryst. from EtOH. M.p.  $225^\circ$ .  $[\alpha]_D^{20} + 147.5^\circ$ .

Barger, Schlittler, *Helv. Chim. Acta*, 1932, 15, 381.

Barger, Girardet, *Helv. Chim. Acta*, 1931, 14, 481.

Aston, *J. Chem. Soc.*, 1910, 97, 1381.

## Pulcheremodin

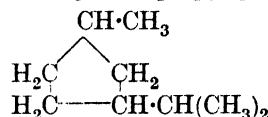
$C_{15}H_{10}O_5$  MW, 270

From root of *Rumex pulcher*, Linn. Orange needles. M.p.  $251^\circ$ . Readily sol. EtOH, Et<sub>2</sub>O, Py. Sol.  $CHCl_3$ ,  $Me_2CO$ , AcOH. Mod. sol. MeOH,  $CS_2$ . Spar. sol.  $CCl_4$ ,  $C_6H_6$ .  $H_2SO_4 \rightarrow$  red col.

*Triacetate*: yellow needles. M.p.  $194^\circ$ . Sol. EtOH, AcOH,  $C_6H_6$ .  $H_2SO_4 \rightarrow$  carmine red col.

Emmanuel, *Chem. Zentr.*, 1918, I, 564.

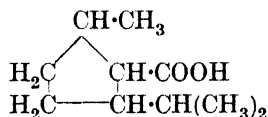
## Pulegan (1-Methyl-3-isopropylcyclopentane)



$C_9H_{18}$  MW, 126

Oil with terpene odour. B.p.  $142-4^\circ$ .  $D_4^{20} 0.7730$ .  $n_D^{20} 1.4236$ .

Wallach, *Ann.*, 1912, 392, 58.

Puleganic Acid (*Dihydropulegenic acid*, 1-methyl-3-isopropylcyclopentane-2-carboxylic acid)

$C_{10}H_{18}O_2$  MW, 170

Colourless viscous oil. F.p.  $-18$  to  $-19^\circ$ . B.p.  $152^\circ/25$  mm.,  $139^\circ/12$  mm.  $D_4^{20} 0.9642$ .  $n_D^{20} 1.4524$ .  $[\alpha]_D^{20} - 0.36^\circ$ .

*Me ester*:  $C_{11}H_{20}O_2$ . MW, 184. B.p.  $91^\circ/13$  mm.

*Et ester*:  $C_{12}H_{22}O_2$ . MW, 198. B.p.  $145^\circ/4$  mm.  $D_4^{11.8} 0.9178$ .  $n_D^{11.8} 1.4405$ .

*Chloride*:  $C_{10}H_{17}OCl$ . MW, 188.5. B.p.  $89-90^\circ/11$  mm.

*Amide*:  $C_{10}H_{19}ON$ . MW, 169. Cryst. from EtOH.Aq. or AcOEt-ligroin. M.p.  $150-1^\circ$ .  $[\alpha]_D^{20} + 4.8^\circ$  in MeOH.

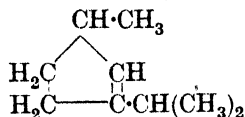
*Nitrile*:  $C_{10}H_{17}N$ . MW, 151. B.p.  $103^\circ/13$  mm.  $D_4^{18.2} 0.8814$ .  $n_D^{18.2} 1.4475$ .

*Anilide*: cryst. from EtOH.Aq. M.p.  $149-50^\circ$ .

Rupe, Schäfer, *Helv. Chim. Acta*, 1928, 11, 467.

Wallach, *Ann.*, 1918, 414, 237.

## Pulegene (3-Methyl-1-isopropylcyclopentene)



$C_9H_{16}$  MW, 124

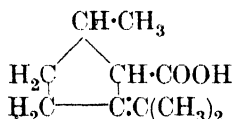


Oil. B.p. 138–9°, 39–41°/16 mm.  $D_4^{22}$  0.791.  $n_D^{22}$  1.4380.

Nitrosochloride: cryst. M.p. 74–5°.

Wallach, Collmann, Thede, *Ann.*, 1903, 327, 131.

**Pulegenic Acid** (1-Methyl-3-isopropylidene-cyclopentane-2-carboxylic acid)



$C_{10}H_{16}O_2$

MW, 168

Pale yellow oil. B.p. 144–6°/12 mm.  $D_4^{20}$  1.0050.  $n_D^{20}$  1.4754.  $[\alpha]_D^{20} + 48.18^\circ$ . MeOH-HCl  $\rightarrow$  hydrochloride of Me ester. Aniline at 200°  $\rightarrow$  pulegene.

Me ester:  $C_{11}H_{18}O_2$ . MW, 182. B.p. 98–101°/10 mm.  $D_4^{20}$  0.97.  $n_D^{20}$  1.4665. Hydrochloride: m.p. 15–16°. B.p. 114°/12 mm.

Amide:  $C_{10}H_{17}ON$ . MW, 167. Needles from  $H_2O$  or EtOH.Aq. M.p. 123°. Very sol. EtOH, Et<sub>2</sub>O.  $[\alpha]_D^{18} + 29.05^\circ$  in MeOH. Red.  $\rightarrow$  amide of puleganic acid.

Nitrile:  $C_{10}H_{15}N$ . MW, 149. B.p. 218–20°.  $n_D^{22}$  1.47047.

Anilide: needles from Et<sub>2</sub>O–pet. ether. M.p. 124°. B.p. 200°/10 mm. Very sol. EtOH, Et<sub>2</sub>O. Spar. sol. pet. ether.

p-Toluidide: needles from Et<sub>2</sub>O–pet. ether. M.p. 143°.

Rüpe, Schäfer, *Helv. Chim. Acta*, 1928, 11, 466.

Wallach, *Ann.*, 1918, 414, 242.

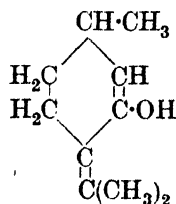
**$\beta$ -Pulegenic Acid** (Isopulegenic acid).

Differs from pulegenic acid in position of double bond. Oil. B.p. 142–5°/11 mm.  $D_4^{20}$  0.9975.  $n_D$  1.4747.  $[\alpha]_D^{22} + 32.7^\circ$  in Et<sub>2</sub>O.

Amide:  $C_{10}H_{17}ON$ . MW, 167. Needles from MeOH.Aq. M.p. 152°. Red.  $\rightarrow$  amide of puleganic acid.

See last reference above.

**Pulegenol** (Enol form of pulegone,  $\Delta^2, 4^{(8)}$ -p-menthadienol-3)



$C_{10}H_{16}O$

MW, 152

Oil. B.p. 85°/6 mm.  $D_4^{13}$  0.916.  $n_D^{20}$  1.48312.  $[\alpha]_D + 24.6^\circ$ . Dist. in steam or action of alkalis  $\rightarrow$  pulegone.

Acetate: oil.

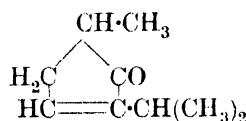
Benzoate: cryst. M.p. 230°.

Et ether:  $C_{12}H_{20}O$ . MW, 180. B.p. 97–97.5°/12 mm.  $D_4^{20}$  0.9047.

Grignard, Blanchon, *Bull. soc. chim.*, 1931, 49, 23.

Grignard, Savard, *Bull. soc. chim. Belg.*, 1927, 36, 97; *Compt. rend.*, 1924, 179, 1573.

**Pulegenone** (4-Methyl-1-isopropylcyclopentenone-5)



$C_9H_{14}O$

MW, 138

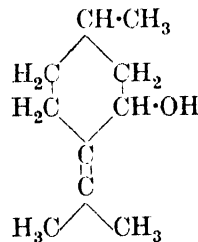
Oil. B.p. 188.5–189°.  $D_4^{20}$  0.9144.  $n_D^{20}$  1.4660.

Oxime: b.p. 237–42°, 123–6°/15 mm. Benzoate: cryst. from MeOH. M.p. 104–5°.

Semicarbazone: cryst. from MeOH. M.p. 183–4°.

Wallach, Grote, *Ann.*, 1919, 418, 50.

**Pulegol** ( $\Delta^2, 4^{(8)}$ -p-Menthenol-3, 1-methyl-4-isopropylidencyclohexanol-3)



$C_{10}H_{18}O$

MW, 154

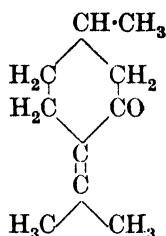
Two forms are described. (i) Obtained by Na–EtOH reduction of pulegone. Needles. M.p. 46–7°. B.p. 209–10°.  $[\alpha]_D - 54.1^\circ$  in EtOH. Hydrogen phthalate: needles from pet. ether. M.p. 212°.  $[\alpha]_D - 86.8^\circ$  in EtOH. (ii) Obtained by reduction of pulegone with aluminium isopropylate. B.p. 91.5°/12 mm.  $D_4^{18}$  0.909.  $n_D^{18}$  1.4714.  $[\alpha]_{D461}^{18} + 80.09^\circ$ .

Doeuvre, Perret, *Bull. soc. chim.*, 1935, 2, 298.

Paolini, *Atti accad. Lincei*, 1919, 28, 190, 236.

**$\beta$ -Pulegomenthol.**

See Neomenthol.

**Pulegone** ( $\Delta^{4(8)}$ -p-Menthenone-3) $C_{10}H_{16}O$ 

MW, 152

In oils from *Mentha pulegium*, *Hedeoma pulegoides*, *Mentha sylvestris*, etc. Colourless oil with pleasant peppermint odour. B.p.  $224^{\circ}$ ,  $151-3^{\circ}/100$  mm.,  $103^{\circ}/17$  mm.  $D_4^{19}$  0.937.  $n_D^{19}$  1.4880.  $[\alpha]_{D}^{20} + 28-23^{\circ}$ . Heat of comb.  $C_v$  1411.6 Cal. Readily purified through  $NaHSO_3$  comp. Ox.  $\rightarrow$  acetone + *d*-2-methyladipic acid.  $NH_2 \cdot NH_2 \rightarrow$  *d*-isopulegone oxime.

Hydrochloride: cryst. from ligroin. M.p.  $24-5^{\circ}$ .

Hydrobromide: cryst. from EtOH.Aq. M.p.  $40-5^{\circ}$ . Very sol. EtOH,  $Et_2O$ .  $[\alpha]_D - 33-88^{\circ}$  in EtOH.

Nitrosite: needles from EtOH. M.p.  $68-9^{\circ}$ . Sol. EtOH, AcOH,  $CHCl_3$ .

Dinitroso deriv.: m.p.  $81.5^{\circ}$ .

2:4-Dinitrophenylhydrazones: red plates from pet. ether. M.p.  $142^{\circ}$ .

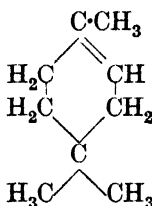
Semicarbazone: prisms from EtOH. M.p.  $174^{\circ}$ . Mod. sol. EtOH.

Phenylsemicarbazone: needles from EtOH. M.p.  $132-3^{\circ}$ .

Doeuvre, Perret, *Bull. soc. chim.*, 1935, 2, 298.

Kon, *J. Chem. Soc.*, 1930, 1616.

Baeyer, Henrich, *Ber.*, 1895, 28, 652.

**Pulenene** (1:4:4-Trimethylcyclohexene) $C_9H_{16}$ 

MW, 124

Liq. with pleasant odour. B.p.  $139.5-140.5^{\circ}$ ,  $36.3-37.3^{\circ}/14$  mm.  $D_4^{18.5}$  0.8032.  $n_D^{23.2}$  1.444. Ox.  $\rightarrow$  2:2-dimethyl-4-acetyl-*n*-valeric acid.  $H_2SO_4 \rightarrow$  reddish-blue col.

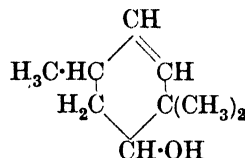
Nitroschloride: rhombohedra from EtOH, needles from AcOEt. M.p.  $118-22^{\circ}$ .

Dict. of Org. Comp.—III.

Other pulenenes are described in the literature. Little data is given and they are of doubtful purity.

Auwers, Lange, *Ann.*, 1915, 409, 167.

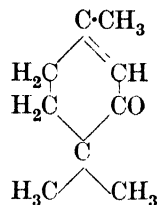
Wallach, Kempe, *Ann.*, 1903, 329, 89.

 $\beta\gamma$ -Pulenenol (3:3:6-Trimethylcyclohexenol-4) $C_9H_{16}O$ 

MW, 140

Oil with peppermint odour. B.p.  $189^{\circ}/754$  mm.,  $82-5^{\circ}/15$  mm.  $D_4^{18.5}$  0.9209.  $n_D^{18.5}$  1.47398.

Auwers, Hessenland, *Ber.*, 1908, 41, 1807.

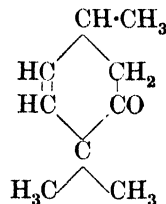
 $\alpha\beta$ -Pulenenone (1:4:4-Trimethylcyclohexenone-3) $C_9H_{14}O$ 

MW, 138

Oil with pleasant odour. B.p.  $208^{\circ}/753$  mm.,  $86-8^{\circ}/15$  mm.  $D_4^{16.5}$  0.9317.  $n_D^{16.5}$  1.47958. Na-EtOH  $\rightarrow$  pulenol.

Semicarbazone: prisms from MeOH. M.p.  $200-1^{\circ}$ . Very sol. hot MeOH. Mod. sol. cold MeOH.

Auwers, Hessenland, *Ber.*, 1908, 41, 1812.

 $\beta\gamma$ -Pulenenone (3:3:6-Trimethylcyclohexenone-4) $C_9H_{14}O$ 

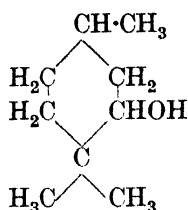
MW, 138

Oil with peppermint odour. B.p.  $172-4^{\circ}$ ,  $63-5^{\circ}/16$  mm.  $D_4^{17.7}$  0.9055.  $n_D^{17.7}$  1.45582.

Dichloride: oil. B.p.  $121-122.5^{\circ}/13$  mm.  $D_4^{22}$  1.2008.  $n_D^{22}$  1.50002.

Semicarbazone: needles from MeOH.Aq. or  $C_6H_6$ -ligroin. M.p.  $127^{\circ}$ .

See previous reference.

**Pulenol** (2 : 2 : 5-Trimethylcyclohexanol) $C_9H_{18}O$ 

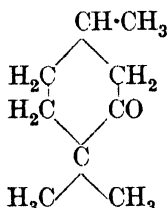
MW, 142

Oil. B.p. 187–9°, 90–2°/23 mm.  $D^{20}_D$  0.8955.  $n^{20}_D$  1.4569.  $CrO_3 \rightarrow$  pulenone.

Phenylurethane: cryst. from EtOH. Aq. M.p. 92° (82–5°).

Auwers, Hessenland, *Ber.*, 1908, **41**, 1814.

Wallach, Kempe, *Ann.*, 1903, **329**, 87.

**Pulenone** (2 : 2 : 5-Trimethylcyclohexanone) $C_9H_{16}O$ 

MW, 140

d.

B.p. 183°. Spar. sol.  $H_2O$ .  $D^{21}_D$  0.8925.  $n^{21}_D$  1.44506. Strongly dextrorotatory.

Oxime: needles. M.p. 94–5°. B.p. 117°/12 mm.

Semicarbazone: m.p. 169–70°.

dl.

B.p. 182–4°, 90–2°/39 mm.  $D^{24}_D$  0.8871.  $n^{24}_D$  1.4432.

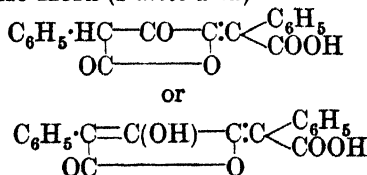
Oxime: cryst. from EtOH. M.p. 93–5°.

Semicarbazone: m.p. 176–7° (169–70°).

See first reference above and also

Wallach, Kempe, *Ann.*, 1903, **329**, 86.

Cornubert, Humeau, *Bull. soc. chim.*, 1931, **49**, 1469.

**Pulvinic Acid** (*Pulvic acid*) $C_{18}H_{12}O_5$ 

MW, 308

Orange powder from  $Et_2O$  or  $CHCl_3$ , prisms from  $C_6H_6$ , yellow cryst. + 1MeOH from MeOH,

yellowish-red cryst. + 1EtOH from EtOH. M.p. 216–17°. Very sol. EtOH, hot AcOH. Sol.  $H_2O$  and pptd. by acids. Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Heat. above m.p., or with  $Ac_2O$  or  $CH_2COCl \rightarrow$  lactone. Ox.  $\rightarrow$  oxalic and benzoylformic acids.

Me ester: see Vulpinic Acid.

Et ester:  $C_{20}H_{16}O_5$ . MW, 336. Yellow tablets from EtOH. M.p. 127–8°. Heat above m.p.  $\rightarrow$  lactone. Acetyl: needles. M.p. 143–4°.

Me ether: needles from EtOH. Prisms from AcOH. M.p. 150–1°.

Propyl ester:  $C_{21}H_{18}O_5$ . MW, 350. Yellow needles or plates from  $CHCl_3$ . M.p. 134°. Very sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH. Me ether: needles. M.p. 121–2°.

Amide:  $C_{18}H_{13}O_4N$ . MW, 307. Yellow prisms from  $C_6H_6$ , yellow plates from AcOH. Sinters at 220–1°, m.p. 226°. Very sol.  $Me_2CO$ . Mod. sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. EtOH. Insol.  $H_2O$ .  $NH_4$  salt: needles. M.p. 218°. Very sol. EtOH. Spar. sol. cold  $H_2O$ .

Me ether: cryst. from MeOH. M.p. 216–17°.

Methylamide: plates from EtOH- $C_6H_6$ . M.p. 237°.

Dimethylamide: prisms. M.p. 211°.

Nitrile:  $C_{18}H_{11}O_3N$ . MW, 289. Reddish-yellow needles from EtOH. Sinters at 190°, m.p. 193–4°. Acetyl: yellow needles from EtOH. M.p. 141–2°. Very sol.  $Et_2O$ . Benzoyl: pale yellow needles from EtOH. M.p. 168–168.5°. Insol.  $H_2O$ .

Anilide: cryst. from AcOH. M.p. 187–8°.

1-Naphthylamide: reddish-yellow plates from toluene. M.p. 211–12°.

2-Naphthylamide: reddish-yellow cryst. from toluene. M.p. 192°.

Asano, Yameda, *Ber.*, 1935, **68**, 1569.

Koller, Pfeiffer, *Monatsh.*, 1933, **62**, 164.

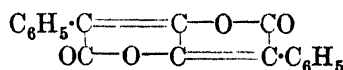
Karrer, Gehrekens, Heuss, *Helv. Chim.*

*Acta*, 1926, **9**, 456.

Mazza, *Chem. Zentr.*, 1926, II, 1037.

Volhard, *Ann.*, 1894, **282**, 14.

Schenck, *ibid.*, 39.

**Pulvinic Acid Lactone** (*Pulvic acid lactone*) $C_{18}H_{10}O_4$ 

MW, 290

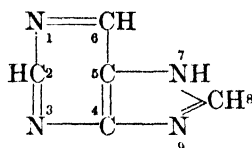
Found in *Sticta aurata* Ach, *Candelaria medians*, etc. Pale yellow needles from  $C_6H_6$  or AcOH. M.p. 222–4°. Sol. hot  $CHCl_3$ ,  $C_6H_6$ , AcOH,  $Me_2CO$ . Spar. sol. hot EtOH. Insol.  $H_2O$ . Insol. cold aq. alkalis. Sublimes in

needles. Hyd.  $\rightarrow$  pulvinic acid.  $\text{NH}_4\text{OH}$   $\rightarrow$   $\text{NH}_4$  salt of pulvinic acid amide.  $\text{KOH-MeOH}$   $\rightarrow$  vulpinic acid. Aniline  $\rightarrow$  pulvinic acid anilide.

A second lactone of pulvinic acid is described in the literature. Cryst. from  $\text{AcOH}$ . M.p.  $124-5^\circ$ .

See previous references.

### Purine



$\text{C}_5\text{H}_4\text{N}_4$

MW, 120

Microscopic needles from  $\text{EtOH}$  or toluene. M.p.  $216-17^\circ$ . Very sol.  $\text{H}_2\text{O}$ , hot  $\text{EtOH}$ . Mod. sol. hot  $\text{AcOEt}$ ,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Amphoteric. Very stable to ox. agents. Gives cryst. hydrochloride and cryst. metallic salts. Forms insol. Zn salt.

$\text{B, HNO}_3$ : cryst. from  $\text{H}_2\text{O}$ . M.p.  $205^\circ$  decomp. Very sol. hot  $\text{H}_2\text{O}$ . Spar. sol. hot  $\text{EtOH}$ .

*Picrate*: plates from hot  $\text{H}_2\text{O}$ . M.p.  $208^\circ$ .

*7-Me*:  $\text{C}_6\text{H}_6\text{N}_4$ . MW, 134. Needles from  $\text{EtOH}$ . M.p.  $184^\circ$ . Insol. alkalis. Sol.  $\text{H}_2\text{O}$ , boiling  $\text{EtOH}$ . Spar. sol.  $\text{C}_6\text{H}_6$ . Heat of comb.  $\text{C}_v$  820.6 Cal. Hydrochloride, nitrate, sulphate, very sol.  $\text{H}_2\text{O}$ . Forms cryst. Pt, Ag, Au salts.  $\text{HgCl}_2$  salt: prisms from  $\text{H}_2\text{O}$ . M.p.  $252^\circ$ . *Methiodide*: yellow needles from  $\text{MeOH}$ . M.p.  $231-2^\circ$ . Very sol.  $\text{H}_2\text{O}$ .

*9-Me*: needles from toluene. M.p.  $162-3^\circ$ . Very sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Sol. toluene. Sublimes.

Montequi, *Chem. Abstracts*, 1927, **21**, 3353.

Fischer, *Ber.*, 1899, **32**, 493; 1898, **31**, 2564.

### Purpuric Acid

$\text{C}_8\text{H}_5\text{O}_6\text{N}_5$

MW, 267

Not known in free state. Acidification of salt sols.  $\rightarrow$  uramil and alloxan.

$\text{NH}_4$  salt: see Murexide.

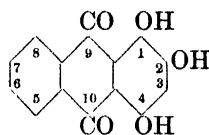
$\text{A, CH}_3\cdot\text{NH}_2$ : cryst. +  $1\text{H}_2\text{O}$ . Anhyd. at  $110^\circ$ , decomp. at  $210^\circ$ . Mod. sol.  $\text{H}_2\text{O}$ . Spar. sol. Py. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

$\text{A, CH}_3\cdot\text{CH}_2\cdot\text{NH}_2$ : red prisms +  $1\text{H}_2\text{O}$ . Anhyd. at  $110^\circ$ , decomp. at  $205^\circ$ .

Hantzsch, Robison, *Ber.*, 1910, **43**, 92.

Möhlau, Litter, *J. prakt. Chem.*, 1906, **73**, 449.

### Purpurin (1 : 2 : 4-Trihydroxyanthraquinone)



$\text{C}_{14}\text{H}_8\text{O}_5$

MW, 256

As glucoside in madder root. Long orange-red or orange-yellow needles +  $\text{H}_2\text{O}$  from  $\text{EtOH.Aq.}$ ; dark red needles from  $\text{EtOH}$ . Anhyd. at  $100^\circ$ , m.p.  $259^\circ$  ( $253^\circ$ ). Very sol. boiling  $\text{AcOH}$ , boiling  $\text{C}_6\text{H}_6$ ,  $\text{EtOH}$ . Spar. sol. boiling  $\text{H}_2\text{O}$ . Sol.  $\text{Et}_2\text{O}$   $\rightarrow$  deep yellow fluorescent sol. Sol.  $\text{CS}_2$ . Sol.  $\text{H}_2\text{SO}_4$   $\rightarrow$  red. sol. Sol. alkalis  $\rightarrow$  red. sols. Ox.  $\rightarrow$  phthalic acid.  $\text{H}_2\text{SO}_4$   $\rightarrow$  purpurin-3-sulphonic acid. Red.  $\rightarrow$  purpuroxanthin. Br  $\rightarrow$  3-bromo deriv.  $\text{NH}_3$   $\rightarrow$  4-aminopurpuroxanthin. Gives cryst. Na, K, Pb salts.

*2-Me ether*:  $\text{C}_{15}\text{H}_{10}\text{O}_5$ . MW, 270. Brick-red needles from  $\text{C}_6\text{H}_6$ . M.p.  $232-3^\circ$  ( $240-2^\circ$ ). Insol. carbonate sols. Sol. alkalis  $\rightarrow$  bluish-red col. Very sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ . *1-Acetyl*: yellow needles from  $\text{Me}_2\text{CO}$ . M.p.  $224-5^\circ$ . *1:4-Di-acetyl*: yellow needles. M.p.  $170-2^\circ$ .

*2:4-Di-Me ether*:  $\text{C}_{16}\text{H}_{12}\text{O}_5$ . MW, 284. Orange needles. M.p.  $186-9^\circ$ . *Acetyl*: lemon-yellow needles from  $\text{EtOH}$ . M.p.  $189-90^\circ$ .

*2-Acetyl*: orange needles from  $\text{EtOH}$ . M.p.  $179-80^\circ$ . Sol. hot  $\text{C}_6\text{H}_6$ , hot  $\text{AcOH}$ . Spar. sol.  $\text{EtOH}$  to fluorescent sol.

*Triacetyl*: pale yellow needles. Sinters at  $193^\circ$ , m.p.  $198-200^\circ$ .

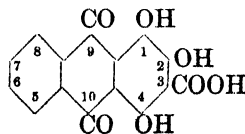
du Pont, U.S.P., 1,985,452, (*Chem. Zentr.*, 1935, II, 439).

Newport Chem. Corp., U.S.P., 1,790,932, (*Chem. Abstracts*, 1931, **25**, 1539).

Marshall, *J. Chem. Soc.*, 1931, 3206.

Perkin, Storey, *J. Chem. Soc.*, 1928, 238.

### Purpurin-3-carboxylic Acid (*Pseudopurpurin*, 1 : 3 : 4-trihydroxyanthraquinone-2-carboxylic acid)



$\text{C}_{15}\text{H}_8\text{O}_7$

MW, 300

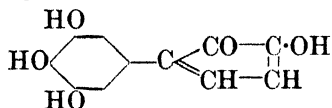
In madder root. Cryst. M.p.  $222-4^\circ$  decomp. Sol. alkalis,  $\text{H}_2\text{SO}_4$ . Mod. sol. boiling  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ . Sol. hot  $\text{H}_2\text{O}$   $\rightarrow$  orange-red sol.

Insol. cold  $\text{H}_2\text{O}$ , EtOH. At  $180-95^\circ \rightarrow$  purpurin.  $\text{Ac}_2\text{O}$  at  $180^\circ \rightarrow$  triacetylurpurin.

Baeyer, D.R.P., 272,301, (*Chem. Zentr.*, 1914, I, 1474).

Liebermann, Plath, *Ber.*, 1877, 10, 1618.

### Purpurogallin



$\text{H}_8\text{O}_5$

MW, 220

In gall of *Dryophanta divisa* as glucoside. Yellow or dark red needles from AcOH. M.p.  $274^\circ$  (rapid heat.). Decomp. without melting when slowly heated. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Mod. sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ . Reduces Au, Ag and alk. Cu sols. Characteristic col. with alkalis, orange-red  $\rightarrow$  blue  $\rightarrow$  green  $\rightarrow$  dark yellow.  $\text{H}_2\text{SO}_4 \rightarrow$  orange-red col. Gives spar. sol. Ba salt.

*Tetra-acetyl*: colourless plates from EtOH or  $\text{C}_6\text{H}_6$ . M.p.  $182-3^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

*Tribenzoyl*: colourless prisms. M.p.  $212-13^\circ$ . Insol. EtOH.

*Tri-Me ether*:  $\text{C}_{14}\text{H}_{14}\text{O}_5$ . MW, 262. Orange needles from AcOEt. M.p.  $174-7^\circ$ . Spar. sol. EtOH. Insol. aq. alkalis. *Acetyl*: needles from EtOH. M.p.  $140-3^\circ$ . Spar. sol. EtOH.

*Tetra-Me ether*:  $\text{C}_{15}\text{H}_{16}\text{O}_5$ . MW, 276. Prisms from EtOH. M.p.  $93-4^\circ$ . Sol. EtOH.

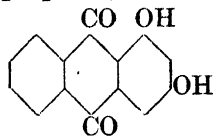
Evans, Dehn, *J. Am. Chem. Soc.*, 1930, 52, 3649.

Wilstätter, Heiss, *Ann.*, 1923, 433, 17.

Nierenstein, *J. Chem. Soc.*, 1919, 115, 1331.

Perkin, Steven, *J. Chem. Soc.*, 1903, 83, 194.

### Purpuroxanthin (1 : 3-Dihydroxyanthraquinone, xanthopurpurin)



$\text{C}_{14}\text{H}_8\text{O}_4$

MW, 240

Yellow needles or leaflets. M.p.  $268-70^\circ$  ( $264^\circ$ ). Sol.  $\text{Me}_2\text{CO}$ , AcOH,  $\text{PhNO}_2$ . Mod. sol. EtOH,  $\text{C}_6\text{H}_6$ . Sol. alkalis  $\rightarrow$  red. sols. Sublimes. Alk. fusion or  $\text{H}_2\text{SO}_4 + \text{MnO}_2 \rightarrow$  purpurin. Zn dust dist.  $\rightarrow$  anthracene.  $\text{HNO}_3 \rightarrow$  phthalic acid.

1-*Acetyl*: orange-yellow needles from MeOH. M.p.  $231-5^\circ$ . Very sol.  $\text{Me}_2\text{CO}$ .

3-*Acetyl*: yellow needles from EtOH. M.p.  $144^\circ$ .

*Diacetyl*: pale yellow needles. M.p.  $183-4^\circ$ .

1-*Me ether*:  $\text{C}_{15}\text{H}_{10}\text{O}_4$ . MW, 254. Yellow leaflets from  $\text{Me}_2\text{CO}$ . M.p.  $311-13^\circ$ . *Acetyl*: yellow leaflets from EtOH. M.p.  $154-5^\circ$ .

3-*Me ether*: pale yellow needles from AcOH. M.p.  $193^\circ$ .

*Di-Me ether*:  $\text{C}_{16}\text{H}_{12}\text{O}_4$ . MW, 268. Yellow needles. M.p.  $151-3^\circ$ .

*Di-phenyl ether*: 1 : 3-diphenoxyanthraquinone. Yellow needles from AcOH. M.p.  $167^\circ$ .

Perkin, Story, *J. Chem. Soc.*, 1929, 1399.

### Purpuroxanthin-2-carboxylic Acid.

See Munjistin.

### Putrescine (Tetramethylenediamine, 1 : 4-diaminobutane)



$\text{C}_4\text{H}_{12}\text{N}_2$

MW, 88

Product of decomp. of excreta, dead animal matter, etc. Cryst. with strong odour. M.p.  $27-8^\circ$  ( $24^\circ$ ). B.p.  $158-9^\circ$ .  $D_4^{25}$  0.877. Sol.  $\text{H}_2\text{O}$ . Absorbs  $\text{CO}_2$ .  $k = 5.1 \times 10^{-4}$  at  $25^\circ$ . Salts are not poisonous.

*B,2HCl*: needles from  $\text{H}_2\text{O}$ . Does not melt below  $290^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Insol. MeOH.

*B,2HAuCl\_4*: cryst. +  $2\text{H}_2\text{O}$ . M.p.  $235-40^\circ$ .

*B,H\_2PtCl\_6*: darkens at  $230^\circ$ . Does not melt below  $275^\circ$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Insol. EtOH.

N : N' - *Diacetyl*: m.p.  $137^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH.

*Monobenzoyl*: colourless viscous oil. B.p.  $176-8^\circ/0.1$  mm. *Hydrochloride*: m.p.  $167^\circ$ .

N' - *Acetyl*: m.p.  $143^\circ$ . *Picrate*: m.p.  $168-70^\circ$ .

N : N' - *Dibenzoyl*: m.p.  $177^\circ$ .

N : N' - *Di-m-nitrobenzoyl*: m.p.  $246^\circ$ .

*Picrate*: pale yellow prisms. Turns brown at  $250^\circ$ . Decomp. at  $250-5^\circ$ .

N : N' - *Di-Me*:  $\text{C}_8\text{H}_{16}\text{N}_2$ . MW, 116. B.p.  $168^\circ$ .

*Di-m-nitrobenzoyl*: cryst. from MeOH. M.p.  $118^\circ$ .

N : N' - *Tetra-Me*:  $\text{C}_8\text{H}_{20}\text{N}_2$ . MW, 144. Colourless liq. B.p.  $168^\circ$ . Misc. with hot  $\text{H}_2\text{O}$ . Sol. EtOH,  $\text{Et}_2\text{O}$ .  $D_4^{18.9}$  0.8041.  $n_D^{18.9}$  1.4316. Volatile in steam. *B,2HCl*: prisms from EtOH. M.p.  $273^\circ$  decomp. *B,2HAuCl\_4*: yellow prisms from hot  $\text{H}_2\text{O}$ . Sinters at  $200^\circ$ . Decomp. at  $206-7^\circ$ . *B,H\_2PtCl\_6*: prisms +  $2\text{H}_2\text{O}$ . M.p.  $234^\circ$  decomp. *Picrate*: m.p.  $198-9^\circ$ .

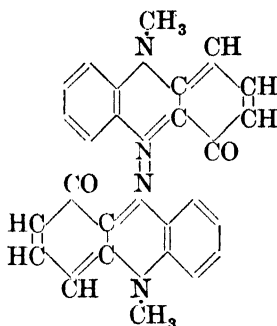
Braun, Pirkernelle, *Ber.*, 1934, 67, 1056.

Keil, *Z. physiol. Chem.*, 1931, 196, 81.

Wrede, Fanselow, Strack, *Z. physiol. Chem.*, 1927, 163, 219.

Keil, *Ber.*, 1926, 59, 2816.

## Pyocyanine

 $C_{26}H_{20}O_2N_4$ 

MW, 420

Pigment of *Bacillus pyocyaneus*. Blue needles from  $CHCl_3$ -pet. ether. M.p.  $133^\circ$ . Sol.  $CHCl_3$ ,  $Me_2CO$ ,  $AcOEt$ , hot  $H_2O$ . Insol.  $Et_2O$ ,  $C_6H_6$ , pet. ether,  $CCl_4$ ,  $CS_2$ . Hyd.  $\rightarrow$  2 mols. hemipyocyanine. Acetylation or benzylation  $\rightarrow$  hemipyocyanine acetate or benzoate. Increases respiration of living cells.

Perchlorate: red needles. M.p.  $221-3^\circ$  decomp.

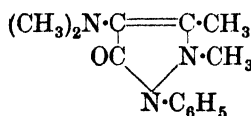
Picrate: red leaflets + 2MeOH from MeOH, or violet leaflets + 1EtOH from EtOH. M.p.  $194-5^\circ$  decomp.

Picrolonate: reddish-black cryst. + 1EtOH. M.p.  $195-6^\circ$ .

Wrede, Strack, *Z. physiol. Chem.*, 1929, **181**, 58; *Ber.*, 1929, **62**, 2051.

McCombie, Scarborough, *J. Chem. Soc.*, 1923, **123**, 3279.

## Pyramidone (4-Dimethylaminoantipyrene, amidopyrine)

 $C_{13}H_{17}ON_3$ 

MW, 231

Cryst. powder. M.p.  $108^\circ$ . Sol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ .  $FeCl_3 \rightarrow$  bluish-violet col. Antipyretic. Less toxic and more powerful than antipyrene.

$B, HBr$ : pyrobromone. Needles. M.p.  $190^\circ$ . Sol.  $H_2O$ ,  $EtOH$ . Insol.  $Et_2O$ ,  $C_6H_6$ .

$B, HI$ : pyro-iodone. M.p.  $196-7^\circ$ .

Citrate: cryst. from  $EtOH.Aq$ . M.p.  $85^\circ$ .

$B, H, Au(CN)_4$ : plates. M.p.  $183-5^\circ$ .

Methobromide: decomp. at  $212^\circ$ .

Methiodide: decomp. about  $220^\circ$ . Sol.  $H_2O$ .

Picrate: m.p.  $168-70^\circ$ .

Styphnate: yellow needles from  $EtOH$ . M.p.  $191.^\circ$

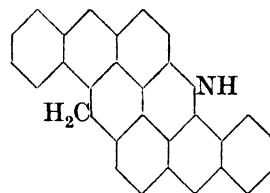
Rodionov, *Bull. soc. chim.*, 1926, **39**, 305. Klebanski, Lemke, *Chem. Abstracts*, 1935, **29**, 6891.

Reuter, U.S.P., 2,005,506, (*Chem. Abstracts*, 1935, **29**, 5130).

I.G., D.R.P., 617,237, (*Chem. Zentr.*, 1935, II, 3949).

Knorr, Stolz, *Ann.*, 1896, **293**, 66.

## Pyranthridine

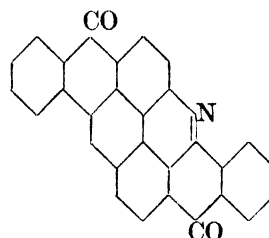
 $C_{29}H_{17}N$ 

MW, 379

Reddish-brown leaflets from xylene. M.p.  $370^\circ$ . Conc.  $H_2SO_4 \rightarrow$  dark blue salt.

Scholl, Dischendorfer, *Ber.*, 1918, **51**, 441.

## Pyranthridone

 $C_{29}H_{13}O_2N$ 

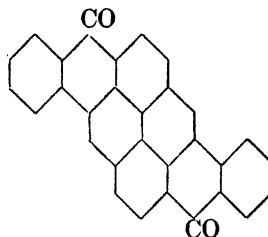
MW, 407

Brownish-yellow needles from quinoline. Does not melt below  $500^\circ$ . Sublimes. Sol. conc.  $H_2SO_4$  with red col. Insol. most org. solvents. Gives violet-blue vat.

See previous reference and also

Scholl, D.R.P., 307,390, (*Chem. Zentr.*, 1918, II, 495).

## Pyranthrone

 $C_{30}H_{14}O_2$ 

MW, 406

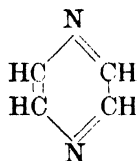
Brown cryst. Sol. hot  $\text{PhNO}_2$ , hot aniline. Sol. conc.  $\text{H}_2\text{SO}_4$  with blue col. Well known orange vat dyestuff.

Scholl, *Ber.*, 1910, **43**, 350; D.R.P., 239,761, (*Chem. Zentr.*, 1911, II, 1498).  
Badische, D.R.P., 175,067, (*Chem. Zentr.*, 1906, II, 1537).  
Gallotti, *Chem. Abstracts*, 1934, **28**, 5063.  
See also Colour Index, No. 1096.

**Pyrantin.**

See under Succinimide.

**Pyrazine (1 : 4-Diazine)**



$\text{C}_4\text{H}_4\text{N}_2$  MW, 80

Prisms from  $\text{H}_2\text{O}$ . M.p.  $47^\circ$ . B.p.  $115.5\text{--}115.8^\circ/768.4$  mm.  $D_4^{60.9}$  1.0311.  $n_D^{60.9}$  1.4953. Sol.  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O. Volatile in steam.

$\text{B}_2\text{H}_2\text{SO}_4$ : m.p.  $136\text{--}7^\circ$ .

$\text{B}_2\text{HAuCl}_4$ : yellow plates from HCl. M.p.  $245^\circ$  decomp.

$\text{B}_2\text{AuCl}_3$ : m.p.  $202\text{--}3^\circ$ .

$\text{B}_2\text{HgCl}_2$ : m.p.  $273^\circ$  decomp.

*Picrate*: yellow needles from EtOH. M.p.  $157^\circ$ .

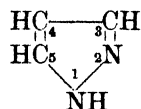
Aston, Peterson, Holowchzk, *J. Am. Chem. Soc.*, 1934, **56**, 153.

Wolff, Marburg, *Ann.*, 1906, **363**, 215.

Stoehr, *J. prakt. Chem.*, 1894, **49**, 392; 1895, **51**, 449.

Gabriel, Pinkus, *Ber.*, 1893, **26**, 2207.

**Pyrazole (1 : 2-Diazole)**



$\text{C}_3\text{H}_4\text{N}_2$  MW, 68

Needles or prisms from ligroin. M.p.  $69.5\text{--}70^\circ$ . B.p.  $186\text{--}8^\circ/757.9$  mm. Sol.  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ .  $k = 3.0 \times 10^{-12}$  at  $25^\circ$ .

$\text{B}_2\text{HCl}$ : hygroscopic needles. M.p.  $104^\circ$ .

$\text{B}_2\text{H}_2\text{SO}_4$ : cryst. from EtOH. M.p.  $134^\circ$ .

$\text{B}_2\text{HNO}_3$ : needles. M.p.  $148^\circ$ .

$\text{B}_2(\text{COOH})_2$ : needles from EtOH-Et<sub>2</sub>O. M.p.  $192^\circ$  decomp.

*N-Me*: see 1-Methylpyrazole.

*N-Phenyl*: see 1-Phenylpyrazole.

*N-Acetyl*: oil. B.p.  $155\text{--}6^\circ/744$  mm. *Picrate*: yellow prisms from EtOH. M.p.  $170\text{--}1^\circ$ .

*N-Benzoyl*: oil. B.p.  $281^\circ/747$  mm.,  $220\text{--}5^\circ/60$  mm.

*Picrate*: yellow needles. M.p.  $160^\circ$ .

v. Pechmann, *Ber.*, 1898, **31**, 2950.

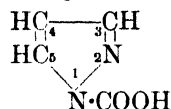
Curtius, Wirsing, *J. prakt. Chem.*, 1894, **50**, 544.

Dains, Harger, *J. Am. Chem. Soc.*, 1918, **40**, 562.

Mingoia, *Chem. Zentr.*, 1931, II, 2324.

Knorr, D.R.P., 74,619.

**Pyrazole-1-carboxylic Acid**



$\text{C}_4\text{H}_4\text{O}_2\text{N}_2$  MW, 112

M.p.  $102\text{--}3^\circ$  decomp. Boil with  $\text{H}_2\text{O} \rightarrow$  pyrazole.

*Et ester*:  $\text{C}_6\text{H}_8\text{O}_2\text{N}_2$ . MW, 140. Oil. B.p.  $213^\circ/741$  mm.,  $120\text{--}3^\circ/42$  mm.

*Amide*:  $\text{C}_4\text{H}_5\text{ON}_3$ . MW, 111. Cryst. from EtOH. M.p.  $136.5^\circ$ .

Mingoia, *Chem. Zentr.*, 1931, II, 2324.

Knorr, *Ber.*, 1895, **28**, 716.

**Pyrazole-3-carboxylic Acid.**

Prisms from  $\text{H}_2\text{O}$ . M.p.  $210\text{--}12^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH. Mod. sol. Et<sub>2</sub>O, AcOH. Insol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

*Me ester*:  $\text{C}_5\text{H}_6\text{O}_2\text{N}_2$ . MW, 126. Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $139\text{--}40^\circ$ . Sol. MeOH. Spar. sol. ligroin.

*N-Me*:  $\text{C}_5\text{H}_6\text{O}_2\text{N}_3$ . MW, 126. Cryst. M.p.  $222^\circ$ . Mod. sol. hot  $\text{H}_2\text{O}$ .

*N-Phenyl*: see 1-Phenylpyrazole-3-carboxylic Acid.

Auwers, Breyhan, *J. prakt. Chem.*, 1935, **143**, 274.

Auwers, Cauer, *J. prakt. Chem.*, 1930, **126**, 187.

v. Pechmann, Burkhard, *Ber.*, 1900, **33**, 3595.

Buchner, Kachumian, *Ber.*, 1902, **35**, 41.

Knorr, Macdonald, *Ann.*, 1894, **279**, 231.

Jowett, Potter, *J. Chem. Soc.*, 1903, **83**, 469.

**Pyrazole-4-carboxylic Acid.**

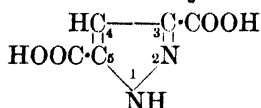
Needles from  $\text{H}_2\text{O}$ . M.p.  $219\text{--}20^\circ$  (decomp. at  $275^\circ$ ). Sol. EtOH. Mod. sol.  $\text{H}_2\text{O}$ , Et<sub>2</sub>O. Sublimes.

*N-Phenyl*: see 1-Phenylpyrazole-4-carboxylic Acid.

Knorr, Laubmann, *Ber.*, 1889, **22**, 180.

Behagel, Buchner, *Ber.*, 1902, **35**, 35.

**Pyrazole-3 : 5-dicarboxylic Acid**



$C_5H_4O_4N_2$  MW, 156

Needles. M.p. 287–90° decomp.  $D^{25}_D$  1.626. Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ . Very spar. sol.  $CHCl_3$ , ligroin.

*Di-Me ester*:  $C_7H_8O_4N_2$ . MW, 184. Plates from  $Et_2O$ . M.p. 151.5°. Sol. EtOH. Spar. sol. ligroin. *N-Me*: needles from ligroin. M.p. 72–73.5°. *N-Acetyl*: plates from EtOH. M.p. 84–5–85°.

*N-Phenyl*: see 1-Phenylpyrazole-3 : 5-dicarboxylic Acid.

Auwers, Cauér, *J. prakt. Chem.*, 1930, **126**, 196.

Gray, *Ber.*, 1900, **33**, 1223.

Knorr, Macdonald, *Ann.*, 1894, **279**, 218 (Footnote).

**Pyrazole-4 : 5-dicarboxylic Acid.**

Needles +  $1H_2O$  from dil.  $HNO_3$ . M.p. 260° decomp. Sol. EtOH. Spar. sol.  $Et_2O$ . Insol.  $CHCl_3$ .

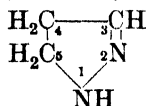
*Di-Me ester*: needles from  $H_2O$ . M.p. 141°.

*N-Phenyl*: see 1-Phenylpyrazole-4 : 5-dicarboxylic Acid.

v. Pechmann, *Ber.*, 1900, **33**, 630.

v. Pechmann, Sell, *Ber.*, 1899, **32**, 2299.

**2-Pyrazoline (4 : 5-Dihydropyrazole)**



$C_3H_6N_2$  MW, 70

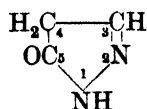
Colourless liq. B.p. 144°.  $D^{20}_D$  1.017.  $n^{20}_D$  1.478. Misc. with  $H_2O$ , EtOH. Volatile in steam and ether.

*N-o-Tolyl*: liq. B.p. 271°/759.5 mm.  $D^{20}_D$  1.084.

*N-p-Tolyl*: needles from EtOH.Aq. M.p. 60.5°. B.p. 281–2°/758 mm.

Auwers, Heimke, *Ann.*, 1927, **458**, 180.

**5-Pyrazolone**



$C_3H_4ON_2$  MW, 84

Needles from toluene. M.p. 165°. Sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ .

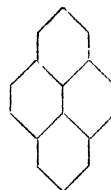
4-Isonitroso : needles from  $H_2O$ . M.p. 180–1°.

Ruhemann, Morrel, *Ber.*, 1895, **28**, 988.

Knorr, *Ber.*, 1896, **29**, 253.

Dains, O'Brien, Johnson, *J. Am. Chem. Soc.*, 1916, **38**, 1510.

**Pyrene**



$C_{16}H_{10}$  MW, 202

Constituent of coal tar. Pale yellow plates by cryst. or sublimation. M.p. 149–50°. Does not boil below 360°. Sol.  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ , toluene. Sols. show blue fluor.  $CrO_3 \rightarrow$  pyrene-quinone  $\rightarrow$  pyrenic acid.

*Picrate*: red needles from EtOH. M.p. 222°. Mod. sol. hot EtOH. Sol.  $CS_2$ ,  $C_6H_6$ .

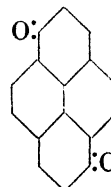
Clar, *Ber.*, 1936, **69**, 1683.

Fleischer, Retze, *Ber.*, 1922, **55**, 3280.

Weitzenböck, *Monatsh.*, 1913, **34**, 221.

Graebe, *Ann.*, 1871, **158**, 285.

**Pyrene-3 : 8-quinone**



$C_{16}H_8O_2$  MW, 232

Yellow needles from  $PhNO_2$ . M.p. 309° (rapid heat.); darkens at 290° (slow heat.). Sol. conc.  $H_2SO_4$  with yellowish-orange col. Sol.  $PhNO_2$ . Spar. sol. EtOH,  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ .

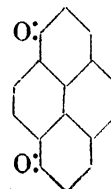
Vollmann, Becker, Corell, Streeck, *Ann.*, 1937, **531**, 1.

Goldschmiedt, *Monatsh.*, 1883, **4**, 310.

Griebe, *Ann.*, 1871, **158**, 295.

See also Scholl, Seer, *Ann.*, 1912, **394**, 125.

**Pyrene-3 : 10-quinone**



$C_{16}H_8O_2$

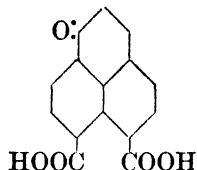
MW, 232



Brownish-red needles from AcOH. M.p. 270°. Sol. conc.  $\text{H}_2\text{SO}_4$  with olive-green col.

See first reference above.

### Pyrenic Acid



$\text{C}_{16}\text{H}_8\text{O}_5$

MW, 268

Pale yellow plates.

$\alpha$ -Me ester:  $\text{C}_{16}\text{H}_{10}\text{O}_5$ . MW, 282. Yellow needles from MeOH. Decomp. at 275°. Sol. boiling EtOH to 0.5%.

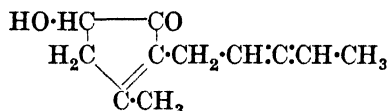
$\beta$ -Me ester: yellowish-green needles from MeOH. Sol. boiling EtOH to 1.06%.

Anhydride:  $\text{C}_{16}\text{H}_6\text{O}_4$ . MW, 250. Needles. Decomp. at 260°.

Imide:  $\text{C}_{15}\text{H}_{11}\text{O}_2\text{N}$ . MW, 237. Yellow plates. Insol. cold alkalis, sol. hot alkalis  $\rightarrow$  red sols.

Bamberger, Philip, *Ann.*, 1887, **240**, 168.  
Langstein, *Monatsh.*, 1910, **31**, 863.

### Pyrethrolone



$\text{C}_{11}\text{H}_{14}\text{O}_2$

MW, 178

Viscous oil. B.p. 115–18°/0.125 mm., 111–12°/0.05 mm. Reduces  $\text{KMnO}_4$  instantly, warm Fehling's, and  $\text{NH}_3\cdot\text{AgNO}_3$  in the cold.

Semicarbazone: cryst. from MeOH. M.p. 200° decomp.

p-Nitrophenylosazone: brown microcryst. powder. Decomp. above 350°.

Acetyl: viscous oil. B.p. 104–5°/0.5 mm.  $[\alpha]_D^{25} - 23.79^\circ$ . Semicarbazone: m.p. 143–5°.

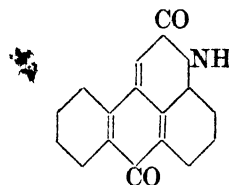
Me ether:  $\text{C}_{12}\text{H}_{16}\text{O}_2$ . MW, 192. B.p. 82–3°/0.25 mm. Semicarbazone: cryst. from MeOH. M.p. 183° decomp.

Et ether:  $\text{C}_{13}\text{H}_{18}\text{O}_2$ . MW, 206. B.p. 102–3° in high vacuo. Semicarbazone: cryst. from MeOH. M.p. 179–80°.

Staudinger, Ruzicka, *Helv. Chim. Acta*, 1924, **7**, 215.

La Forge, Haller, *J. Am. Chem. Soc.*, 1936, **58**, 1061.

### Pyridanthrone (Anthrapyridone)



$\text{C}_{16}\text{H}_9\text{O}_2\text{N}$

MW, 247

Cryst. from  $\text{PhNO}_2$ . M.p. 406–8°. Sol. conc.  $\text{H}_2\text{SO}_4$  with yellow col. and yellow fluor.

N-Me:  $\text{C}_{17}\text{H}_{11}\text{O}_2\text{N}$ . MW, 261. Cryst. from  $\text{PhNO}_2$ . M.p. 267–8°.

Seka, Schreckental, Heilperin, *Monatsh.*, 1929, **53** and **54**, 478.

Höchst, D.R.P., 250,885, (*Chem. Zentr.*, 1912, II, 1319).

Bayer, D.R.P., 209,033, (*Chem. Zentr.*, 1909, I, 1680).

Badische, D.R.P., 216,597, (*Chem. Zentr.*, 1910, I, 68).

### Pyridazine (1 : 2-Diazine, orthodiazine)



$\text{C}_4\text{H}_4\text{N}_2$

MW, 80

Liq. F.p. – 8°. B.p. 208°.  $D_4^{25} 1.1035$ .  $n_D^{25} 1.5231$ . Very sol.  $\text{H}_2\text{O}$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol. pet. ether.  $\text{Na} + \text{EtOH} \rightarrow$  putrescine.

$\text{B}_2\text{AuCl}_3$ : yellow needles from  $\text{H}_2\text{O}$ . M.p. 170° decomp. Insol. cold  $\text{H}_2\text{O}$ .

Chloroaurate: yellow cryst. M.p. 110°.

$\text{B}_2\text{H}_2\text{PtCl}_6$ : orange prisms. Decomp. at 218°. Sol.  $\text{H}_2\text{O}$ .

Picrate: yellow needles from  $\text{H}_2\text{O}$  or EtOH. M.p. 169° decomp.

Wohl, Bernreuther, *Ann.*, 1930, **481**, 12.

Gabriel, *Ber.*, 1909, **42**, 658.

### Pyridine



$\text{C}_5\text{H}_5\text{N}$

MW, 79

Occurs in coffee oil. Extracted in quantity from coal tar. Colourless liq. with characteristic odour. F.p. – 42°. B.p. 115.5°. Forms

azeotropic mixture with 3 mols.  $\text{H}_2\text{O}$ , b.p.  $92-3^\circ$ . Very hygroscopic. Misc. with  $\text{H}_2\text{O}$  in all proportions. Volatile in steam.  $D_4^{25}$  0.97796.  $n_D^{21}$  1.5092.  $k = 2.4 \times 10^{-9}$  at  $25^\circ$ . Heat of comb.  $C_v$  664.7 Cal.,  $C_p$  675.1 Cal. Latent heat of vap. 101.4 Cal. per kilogram at  $115.5^\circ$ . Sp. heat ( $21^\circ$ ) 0.391. Crit. temp. about  $344^\circ$ . F.p. depression 4.97 per kilogram. B.p. elevation 2.69 per kilogram. Red heat  $\rightarrow$  2:4'-dipyridyl. Red.  $\rightarrow$  piperidine.  $\text{H}_2\text{SO}_4 \rightarrow$  3-sulphonic acid.  $\text{NaNH}_2 \rightarrow$  2-aminopyridine.  $\text{MeOH-HCl} \rightarrow$  pyridine methochloride.  $\text{ClCH}_2\text{-COOEt} \rightarrow$  pyridine-betaine hydrochloride. Used for denaturing industrial spirit. Powerful solvent.

$B, \text{HCl}$ : hygroscopic plates from EtOH. M.p.  $82^\circ$ . B.p.  $218-19^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ .

$B, 2\text{HCl}$ : prisms. M.p.  $46-7^\circ$ .

$B, \text{HBr}$ : reddish-yellow plates. M.p.  $213^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ .

$B, \text{HI}$ : cryst. +  $1\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $268^\circ$  decomp.

$B, \text{HClO}_3$ : cryst. from EtOH. M.p.  $147^\circ$  decomp.

$B, (\text{COOH})_2$ : m.p.  $151-2^\circ$ .

Phthalate: cryst. M.p.  $109^\circ$ .

Tartrate: m.p.  $154^\circ$ .

Citrate: m.p.  $123^\circ$ .

3:5-Dinitrobenzoate: yellow needles. M.p.  $162^\circ$ .

$B, \text{HAuCl}_4$ : yellow needles from  $\text{H}_2\text{O}$  or EtOH. M.p.  $329^\circ$  ( $304^\circ$ ). Spar. sol. hot  $\text{H}_2\text{O}$ .

$B, \text{H}_2\text{PtCl}_6$ : reddish-yellow needles. M.p.  $262-4^\circ$  decomp. Very sol.  $\text{H}_2\text{O}$ . Insol. EtOH.

$B, \text{HgCl}_2$ : needles from Py. M.p.  $108^\circ$ .

$B, \text{HgCl}_2$ : needles from  $\text{H}_2\text{O}$  or EtOH. M.p.  $180^\circ$ .

Methiodide: needles or prisms from EtOH or  $\text{Me}_2\text{CO}$ . Very hygroscopic. Yellows at  $100^\circ$ . M.p.  $118^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Sol. EtOH,  $\text{CHCl}_3$ , AcOH,  $\text{Me}_2\text{CO}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ .

Ethobromide: cryst. from EtOH. M.p.  $111-12^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ .

Ethiodide: plates from EtOH- $\text{Et}_2\text{O}$ . M.p.  $90.5^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH, AcOH,  $\text{Me}_2\text{CO}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ .

Propiodide: plates from EtOH. M.p.  $52-3^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH, AcOEt,  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

Isopropiodide: cryst. from EtOH. M.p.  $114-15^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH, AcOEt. Insol.  $\text{Et}_2\text{O}$ .

Phenochloride: needles. M.p.  $105-6^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Sol. EtOH. Insol.  $\text{Et}_2\text{O}$ .

Phenobromide: m.p.  $155^\circ$ .

Pheniodide: pale yellow cryst. M.p.  $207^\circ$ .

Picrate: yellow needles. M.p.  $165-6^\circ$ .

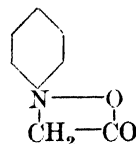
Styphnate: cryst. from EtOH. M.p.  $184.5-185.5^\circ$ .

Ma, Hsia, Sah, *Chem. Abstracts*, 1934, **28**, 3692.

Ferns, Lapworth, *J. Chem. Soc.*, 1912, **101**, 283.

Decker, Kaufmann, *J. prakt. Chem.*, 1911, **84**, 436.

**Pyridine-betaine** (Anhydride or lactone of N-carboxymethylpyridinium hydroxide)



$\text{C}_7\text{H}_7\text{O}_2\text{N}$

MW, 137

Hygroscopic plates +  $1\text{H}_2\text{O}$ . Anhyd. at  $100^\circ$ . M.p.  $150^\circ$  decomp. Very sol.  $\text{H}_2\text{O}$ , hot EtOH. Insol.  $\text{Et}_2\text{O}$ .

$B, \text{HCl}, \text{H}_2\text{O}$ : prisms from EtOH. M.p.  $159^\circ$  decomp. Very sol.  $\text{H}_2\text{O}$ . Insol. EtOH,  $\text{Et}_2\text{O}$ .

$B, \text{HCl}$ : plates from  $\text{H}_2\text{O}$  or EtOH. M.p.  $202-5^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Sol. hot EtOH. Spar. sol. cold EtOH. Insol.  $\text{Et}_2\text{O}$ .

$B, \text{HBr}, \text{H}_2\text{O}$ : plates or prisms from EtOH. Decomp. at  $170^\circ$ .

$B, \text{HBr}$ : plates from EtOH.Aq. Decomp. at  $198-200^\circ$ .

$B, \text{HI}$ : needles from EtOH. Blackens at  $175-80^\circ$ . M.p.  $250-2^\circ$  decomp. Very sol.  $\text{H}_2\text{O}$ . Sol. warm EtOH. Insol.  $\text{Et}_2\text{O}$ .

$B, \text{H}_2\text{SO}_4$ : plates. Decomp. at  $175^\circ$ .

$B, \text{HNO}_3$ : plates from EtOH.Aq. M.p.  $145^\circ$  decomp.

$B, \text{H}_2\text{PtCl}_6$ : orange-red cryst. M.p.  $215^\circ$ . Sol.  $\text{H}_2\text{O}$ . Insol. EtOH.

Chloroaurate: m.p.  $165^\circ$ .

$B, \text{AgNO}_3$ : plates. Decomp. at  $171.5^\circ$ .

$B, (\text{HgCl}_2)_4, \text{HCl}$ : plates +  $1\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $134^\circ$ .

Picrate: yellow prisms from EtOH.Aq. M.p.  $142-3^\circ$ .

Chattaway, Garton, *J. Chem. Soc.*, 1924, **125**, 187.

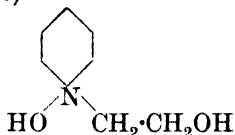
Krüger, *J. prakt. Chem.*, 1891, **43**, 287.

Vongerichten, *Ber.*, 1882, **15**, 1251.

**Pyridine-carboxylic Acid.**

See Picolinic Acid, Nicotinic Acid, and Isonicotinic Acid.

**Pyridine-choline** (N- $\beta$ -Hydroxyethylpyridinium hydroxide)



$C_7H_{11}O_2N$

MW, 141

**Chloride** :  $\beta$ -hydroxyethylpyridinium chloride.  $C_7H_{10}ONCl$ . MW, 159.5. Hygroscopic prisms from EtOH. Very sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .  $Ag_2O \rightarrow$  free base.  $CrO_3 \rightarrow$  pyridine-betaine hydrochloride.  $B_2AuCl_3$  : needles or plates. M.p.  $117^\circ$ . Very sol. boiling  $H_2O$ .  $B_2PtCl_4$  : orange-yellow plates from EtOH. M.p.  $179^\circ$ . Very sol.  $H_2O$ . Insol. cold EtOH.

Schmidt, *Arch. Pharm.*, 1913, 251, 205.

**Pyridine-2 : 3-dicarboxylic Acid.**

See Quinolinic Acid.

**Pyridine-2 : 4-dicarboxylic Acid.**

See Lutidinic Acid.

**Pyridine-2 : 5-dicarboxylic Acid.**

See Isocinchomeronic Acid.

**Pyridine-2 : 6-dicarboxylic Acid.**

See Dipicolinic Acid.

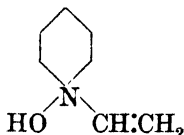
**Pyridine-3 : 4-dicarboxylic Acid.**

See Cinchomeronic Acid.

**Pyridine-3 : 5-dicarboxylic Acid.**

See Dinicotinic Acid.

**Pyridine-neurine** (N-Vinylpyridinium hydroxide)



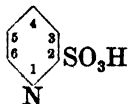
$C_7H_9ON$

MW, 123

**Chloride** : vinylpyridinium chloride.  $C_7H_8NCl$ . MW, 141.5.  $B_2AuCl_3$  : yellow needles from  $H_2O$ . M.p.  $178^\circ$ .  $B_2PtCl_4$  : yellowish-red plates from  $H_2O$ . M.p.  $193^\circ$  decomp.

Schmidt, *Arch. Pharm.*, 1913, 251, 206.

**Pyridine-2-sulphonic Acid**



$C_5H_5O_3NS$

MW, 159

Needles from EtOH. M.p.  $247-8^\circ$ . Very sol.  $H_2O$ .

$NH_4$  salt : m.p.  $274-5^\circ$ .

**Ag salt** : yellow cryst. from  $H_2O$ . M.p.  $290^\circ$  decomp. Very sol. hot  $H_2O$ .

Plazek, Marcinków, *Chem. Abstracts*, 1935, 29, 2535.

Gastel, Wibaut, *Rec. trav. chim.*, 1934, 53, 1031.

Marckwald, Klemm, Trabert, *Ber.*, 1900, 33, 1560.

**Pyridine-3-sulphonic Acid.**

Needles or plates. M.p.  $357^\circ$ . Very sol.  $H_2O$ . Spar. sol. EtOH. Insol.  $Et_2O$ . Dry dist.  $\rightarrow$  3 : 3'-dipyridyl. KOH fusion  $\rightarrow$  3-hydroxypyridine.

$NH_4$  salt : cryst. M.p.  $243^\circ$ .

**Betaine** :  $C_6H_7O_3NS$ . MW, 173. Cryst. from  $H_2O$ . M.p.  $130^\circ$ .

See second reference above and also

I.G., F.P., 685,062, (*Chem. Abstracts*, 1930, 24, 5307).

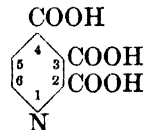
Fischer, *Ber.*, 1882, 15, 62.

**Pyridine-4-sulphonic Acid.**

Needles from EtOH. M.p.  $134-5^\circ$ .

Koenigs, Kinne, *Ber.*, 1921, 54, 1357.

**Pyridine - 2 : 3 : 4 - tricarboxylic Acid**  
(Carbocinchomeronic acid)



$C_8H_5O_6N$

MW, 211

Cryst. +  $1\frac{1}{2}H_2O$  from  $H_2O$ . M.p. anhyd.  $249-50^\circ$  (rapid heat.). Sol. hot  $H_2O$ . Mod. sol. hot EtOH. Insol.  $Et_2O$ ,  $C_6H_6$ . Heat at  $170-80^\circ$  or boiling AcOH  $\rightarrow$  cinchomeronic acid.

**Tri-Me ester** :  $C_{11}H_{11}O_6N$ . MW, 253. Needles. M.p.  $102^\circ$ . **Hydrochloride** : m.p.  $68^\circ$ .

**3 : 4-Di-Et ester** :  $C_{12}H_{13}O_6N$ . MW, 267. M.p.  $118^\circ$ . Sol. most ord. org. solvents. **Hydrochloride** : m.p.  $142^\circ$ .

**Tri-Et ester** :  $C_{14}H_{17}O_6N$ . MW, 295. B.p.  $300-5^\circ$ . **Hydrochloride** : m.p.  $61^\circ$ .

Mumm, Hüneke, *Ber.*, 1918, 51, 159.

Eckert, Loria, *Monatsh.*, 1917, 38, 244.

**Pyridine - 2 : 3 : 5 - tricarboxylic Acid**  
(Carbodinicotinic acid).

Cryst. +  $1\frac{1}{2}H_2O$  from  $H_2O$ . M.p.  $323^\circ$ . Sol. hot  $H_2O$ , EtOH.

Weber, *Ann.*, 1887, 241, 11.

**Pyridine-2 : 3 : 6-tricarboxylic Acid.**

Needles or plates +  $2H_2O$  from  $H_2O$  or EtOH.Aq. M.p.  $130^\circ$ . Very sol.  $H_2O$ , EtOH.Aq.

Spar. sol. EtOH, Et<sub>2</sub>O, AcOH. Heat above m.p.  $\rightarrow$  pyridine-2 : 5-dicarboxylic acid. Gives cryst. spar. sol. Ca salt.

Eckert, Loria, *Monatsh.*, 1917, **38**, 241.

Miller, *Ber.*, 1891, **24**, 1916.

### Pyridine-2 : 4 : 5-tricarboxylic Acid.

See Berberonic Acid.

### Pyridine-3 : 4 : 5-tricarboxylic Acid. ( $\beta$ -Carbocinchomeric acid).

Cryst. in leaflets + 3H<sub>2</sub>O from H<sub>2</sub>O. Chars at 261°. Sol. hot H<sub>2</sub>O.

Weber, *Ann.*, 1887, **241**, 16.

### Pyridinoanthracene.

See Anthraquinoline.

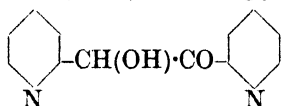
### Pyridinoanthraquinone.

See Anthraquinolinequinone.

### Pyridinostyrene.

See Stilbazole.

### Pyridoin ( $\alpha$ -Hydroxy- $\beta$ -keto-dipyridylethane)



C<sub>12</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub> MW, 214

Yellow needles from EtOH. M.p. 156°. Sol. hot EtOH, Me<sub>2</sub>CO, AcOEt, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. Dil. AcOH  $\rightarrow$  red col. Reduces Fehling's.

Harries, Lénárt, *Ann.*, 1915, **410**, 108.

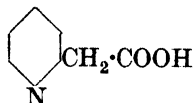
### Pyridone.

See Hydroxypyridine.

### Pyridopyridine.

See Naphthyridine.

### 2-Pyridylacetic Acid



C<sub>7</sub>H<sub>7</sub>O<sub>2</sub>N MW, 137

Cryst. M.p. 98°. Boiling aq. sol.  $\rightarrow$   $\alpha$ -picoline.

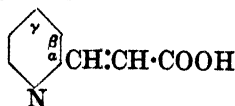
*Me ester* : C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 151. B.p. 123°/12 mm.

*Anilide* : m.p. 134°.

*Picrate* : m.p. 142°.

Oparina, *Chem. Zentr.*, 1935, **I**, 2536.

### 2- $\alpha$ -Pyridylacrylic Acid



C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>N MW, 149

Needles from H<sub>2</sub>O. M.p. 202-3° decomp. Very sol. EtOH. Insol. cold H<sub>2</sub>O. Red.  $\rightarrow$  2- $\alpha$ -pyridylpropionic acid.

*Me ester* : C<sub>9</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 163. *B, HCl* : cryst. from MeOH. M.p. 185-6°.

*Et ester* : C<sub>10</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 177. F.p. 4°. B.p. 161°/25 mm. *B, HAuCl<sub>4</sub>* : yellow needles. M.p. 149°. *B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>* : yellow needles. M.p. 114°.

*B, HCl* : cryst. from EtOH. M.p. 220° decomp.

*B, HBr* : cryst. from AcOH. M.p. 222-3°.

*B, HAuCl<sub>4</sub>* : yellow needles from H<sub>2</sub>O. M.p. 194-5°.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>* : reddish-yellow prisms. M.p. 213° decomp.

*Methobromide* : cryst. from AcOH. M.p. 242°. Very sol. H<sub>2</sub>O. Spar. sol. AcOH. Insol. EtOH.

*Methiodide* : yellow needles from EtOH. Aq. M.p. 219-20° decomp.

Einhorn, *Ann.*, 1891, **265**, 221.

Einhorn, Liebrecht, *Ber.*, 1887, **20**, 1593.

### 2- $\gamma$ -Pyridylacrylic Acid.

Reddish-brown cryst. from AcOEt. M.p. 296° decomp. Very sol. acids, alkalis. Spar. sol. H<sub>2</sub>O. KMnO<sub>4</sub>  $\rightarrow$  isonicotinic acid.

*B, HCl* : brown cryst. + 1H<sub>2</sub>O. M.p. 243-4°. Very sol. H<sub>2</sub>O. Sol. EtOH. Spar. sol. Et<sub>2</sub>O.

*Acetate* : cryst. from AcOH. M.p. 287-8°.

*B, HAuCl<sub>4</sub>* : yellow needles. M.p. 235° decomp. Spar. sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

Alberts, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1285.

Rabe, Kindler, *Ber.*, 1919, **52**, 1842.

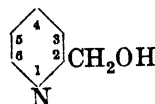
### Pyridylamine.

See Aminopyridine.

### Pyridylbenzene.

See Phenylpyridine.

### 2-Pyridylcarbinol ( $\omega$ -Hydroxy- $\alpha$ -picoline, 2-hydroxymethylpyridine)



C<sub>6</sub>H<sub>7</sub>ON MW, 109

Viscous oil with odour of pyridine. B.p. 112-13°/16 mm. Misc. with H<sub>2</sub>O and most org. solvents.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>* : reddish-yellow needles from EtOH. M.p. 179° decomp. Very sol. H<sub>2</sub>O. Spar. sol. EtOH.

*Picrate*: yellow cryst. from EtOH. M.p. 159°. Spar. sol. cold EtOH.

Harries, Lénárt, *Ann.*, 1915, **410**, 107.

**3-Pyridylcarbinol** ( $\omega$ -Hydroxy- $\beta$ -picoline, 3-hydroxymethylpyridine).

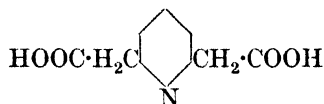
$B,HAuCl_4$ : yellow needles from  $H_2O$ . M.p. 136-7°.

$B_2, H_2PtCl_6$ : brownish-red plates +  $1H_2O$  from  $H_2O$ . M.p. 193-5°.

*Picrate*: yellow needles from  $H_2O$ . M.p. 128°. Very sol. boiling  $H_2O$ ,  $Me_2CO$ .

Dehnel, *Ber.*, 1900, **33**, 3498.

**Pyridyl-2 : 6-diacetic Acid** (2 : 6-Di-[carboxymethyl]-pyridine)



$C_9H_8O_4N$

MW, 195

Cryst. +  $1H_2O$ . M.p. 140°.

*Di-Me ester*:  $C_{11}H_{13}O_4N$ . MW, 223. M.p. 64°.

*Dianilide*: m.p. 198°.  $B_2, H_2PtCl_6$ : M.p. 216°.

Oparina, *Chem. Zentr.*, 1935, **I**, 2536.

**Pyridylethylene.**

See Vinylpyridine.

**Pyridyl Mercaptan.**

See Mercaptopyridine.

**Pyridyl-2-nitramine** (N-Nitro-2-amino-pyridine, 2-nitraminopyridine)



$C_5H_5O_2N_3$

MW, 139

Needles from  $H_2O$ . Decomp. at 184°. Sol. EtOH, hot  $H_2O$ , AcOEt. Sol. dil. alkalis. Spar. sol. strong min. acids. Insol.  $C_6H_6$ , ligroin.

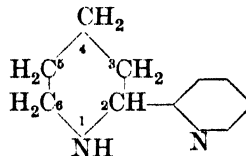
Tschitschibabin, Rasorenow, *J. Russ. Phys.-Chem. Soc.*, 1915, **47**, 1290.

**Pyridyl-3-nitramine** (N-Nitro-3-amino-pyridine, 3-nitraminopyridine).

Small needles from  $H_2O$ . Decomp. at 170-5°. Sol. hot  $H_2O$ . Insol.  $C_6H_6$ . Aq. sol. reacts acid to litmus, neutral to Congo Red. Sol. dil. min. acids.

Tschitschibabin, Kirssanow, *Ber.*, 1927, **60**, 2435.

**2-[2-Pyridyl]-piperidine** (2-[2-Piperidyl]-pyridine)



$C_{10}H_{14}N_2$

MW, 162

B.p. 265-6°/756 mm. Sol.  $H_2O$ , org. solvents.

*Picrate*: prisms from  $H_2O$ . M.p. 187°.

Smith, *J. Am. Chem. Soc.*, 1931, **53**, 281.

**3-[2-Pyridyl]-piperidine.**

See Isonicotine.

**2-[3-Pyridyl]-piperidine.**

See Anabasine.

**3-[3-Pyridyl]-piperidine.**

See Nicotidine.

**4-[3-Pyridyl]-piperidine** (3-[4-Piperidyl]-pyridine).

Oil.

*Picrate*: long needles from  $H_2O$ . M.p. 240° slight decomp.

Smith, *J. Am. Chem. Soc.*, 1931, **53**, 282.

**1-[4-Pyridyl]-piperidine** (4-[1-Piperidyl]-pyridine).

Yellow cryst. M.p. 80°. B.p. 164°/13 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , dil. min. acids. Volatile in steam. Sublimes in vacuo at low temps.

$B,HAuCl_4$ : orange plates. M.p. 161-3°.

*Picrate*: leaflets. M.p. 142°.

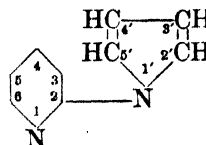
*Methiodide*: yellow cryst. from MeOH. M.p. 159°.

Graf, Lehmann, *J. prakt. Chem.*, 1933, **138**, 242.

**4-[4-Pyridyl]-piperidine.**

See Isonicotine.

**1-[2-Pyridyl]-pyrrole**



$C_9H_8N_2$

MW, 144

M.p. 17°. B.p. 250°/748 mm., 123°/11 mm. Gives intense blue col. with pine splinter moistened with HCl.

*Chloroplatinate*: yellow microcryst. powder +  $2H_2O$ . Slowly decomp. on heating.

*Methiodide*: m.p. 141-2°.

*Picrate*: m.p. 143°.

Tschitschibabin, Bylinkin, *Ber.*, 1923, 56, 1745.

Wibaut, Dingemanse, *Chem. Abstracts*, 1923, 17, 3873.

### 2-[2-Pyridyl]-pyrrole.

Cryst. from ligroin. M.p. 90° (87–8°). Intense blue col. with pine splinter moistened with HCl.

*Methiodide*: m.p. 188°.

*Picrate*: yellow cryst. M.p. 221° (143°).

*N-Me*: see 2:2'-Nicotyrine.

See first reference above and also

Wibaut, *Chem. Abstracts*, 1927, 21, 3362.

Wibaut, *Rec. trav. chim.*, 1926, 45, 657.

### 3-[2-Pyridyl]-pyrrole.

M.p. 132°.

*Methiodide*: m.p. 146°.

*Picrate*: m.p. 193°.

See last two references above.

### 2-[3-Pyridyl]-pyrrole (*Nornicotyrine*).

Needles from ligroin- $C_6H_6$ . M.p. 100–2°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin, H<sub>2</sub>O. EtOH, Et<sub>2</sub>O sols. show blue fluor. FeCl<sub>3</sub> → orange-red col. Gives cryst. K salt.

*B<sub>2</sub>HgCl<sub>2</sub>*: m.p. 178–9°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow needles + 2H<sub>2</sub>O from H<sub>2</sub>O. Decomp. at 150°.

*Methiodide*: pale yellow needles from EtOH. M.p. 170–1°.

*Picrate*: yellow prisms from H<sub>2</sub>O or EtOH. M.p. 202–3° (182°).

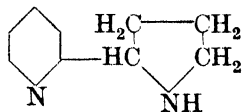
*N-Me*: see 3:2'-Nicotyrine.

Ehrenstein, *Arch. Pharm.*, 1931, 269, 650.

Pictet, *Compt. rend.*, 1903, 137, 861.

Pictet, Crépieux, *Ber.*, 1895, 28, 1909.

### 2-[2-Pyridyl]-pyrrolidine ( $\alpha$ -*Nornicotine*)



$C_9H_{12}N_2$

MW, 148

Oil. B.p. 120°/12 mm. Misc. with H<sub>2</sub>O in all proportions. Sol. most org. solvents.

*Picrate*: cryst. from EtOH. M.p. 166°.

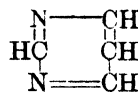
*N-Me*:  $\alpha$ -nicotine.  $C_{10}H_{14}N_2$ . MW, 162. Oil. B.p. 122°/25 mm. Completely misc. with H<sub>2</sub>O and most org. solvents. *Picrate*: cryst. from EtOH. M.p. 169°.

Craig, *J. Am. Chem. Soc.*, 1934, 56, 1146.

### 2-[3-Pyridyl]-pyrrolidine.

See Nornicotine.

### Pyrimidine (1:3-Diazine)



$C_4H_4N_2$

MW, 80

Cryst. mass. M.p. 21°. B.p. 123.5–124°.

Misc. with H<sub>2</sub>O → neutral sol.

*B<sub>2</sub>AuCl<sub>3</sub>*: needles. M.p. 226°.

*Picrate*: yellow needles. M.p. 156°.

Emery, *Ber.*, 1901, 34, 4180.

### Pyrimidole.

See Indolizine.

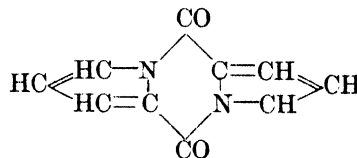
### Pyrobromone.

See under Pyramidone.

### Pyrocatechol.

See Catechol.

### Pyrocoll



$C_{10}H_6O_2N_2$

MW, 186

From gelatine. Plates from AcOH. M.p. 268°. Sol. AcOEt, CHCl<sub>3</sub>. Spar. sol. cold EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, AcOH. Insol. H<sub>2</sub>O. KOH → pyrrole-2-carboxylic acid.

Hale, Hoyt, *J. Am. Chem. Soc.*, 1916, 38, 1065.

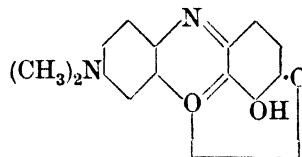
Ciamician, Silber, *Ber.*, 1884, 17, 105.

Weidel, Ciamician, *Monatsh.*, 1880, 1, 279.

### Pyrogallic Acid.

See Pyrogallol.

### Pyrogalline



$C_{14}H_{12}O_3N_2$

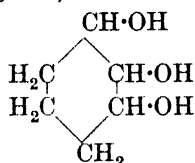
MW, 256

Cryst. with greyish-green reflex from xylene. M.p. 240–1°. Sol. hot H<sub>2</sub>O → bluish-violet sol. Sol. alkalis → reddish-violet sols. H<sub>2</sub>SO<sub>4</sub> → red sol. HCl → blue sol. AcOH → blue sol.

*Me ether*:  $C_{15}H_{14}O_3N_2$ . MW, 270. Metallic green prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 199–200°. Spar. sol. H<sub>2</sub>O → blue sol. Gives cryst. salts with strong acids.

Kehrmann, Beyer, *Ber.*, 1912, 45, 3341.

**Pyrogallitol** (1 : 2 : 3-Trihydroxycyclohexane, hexahydropyrogallol)



$C_6H_{12}O_3$

MW, 132

Three forms are known :

$\alpha$ -.

Needles from AcOEt or  $Me_2CO$ . M.p. 108°.

Tribenzoyl : needles from EtOH. M.p. 142°.

$\beta$ -.

Cryst. from AcOEt. M.p. 124-5°.

Tribenzoyl : cryst. from AcOH. M.p. 184°.

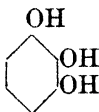
$\gamma$ -.

Cryst. from AcOH. M.p. 148°.

Tribenzoyl : cryst. from EtOH. M.p. 142°.

Lindemann, de Lange, *Ann.*, 1930, **483**, 31.

**Pyrogallol** (Pyrogallic acid, 1 : 2 : 3-trihydroxybenzene)



$C_6H_6O_3$

MW, 126

Plates and needles. M.p. 132.5-133.5°. B.p. 309°, 171.5°/12 mm. Sol. 2.25 parts  $H_2O$  at 13°. Sol. EtOH,  $Et_2O$ . Bitter taste. Heat of comb. 639 Cal. Rapidly absorbs  $O$ , turning brown. Reduces Pt, Au, Ag, Hg salts. Aq. sol. + Pd or enzymes  $\rightarrow$  purpurogallin. Aq. sol. +  $(NH_4)_2CO_3 \rightarrow$  pyrogallol-4-carboxylic acid and pyrogallol-4 : 6-dicarboxylic acid. Red.  $\rightarrow$  1 : 2 : 3-cyclohexanetriol.  $CH_3COCl \rightarrow$  triacetyl deriv. Alk. sol. +  $FeCl_3 \rightarrow$  deep red col.  $H \cdot CHO + HCl \rightarrow$  bright red col., ppt. on boiling. Used as developer in photography, in medicine, and for gas analysis. Is a blood poison.

*Monoacetyl* : viscous oil. B.p. 185°/25 mm.

*Diacetyl* : m.p. 110-11°. Sol. alkalis.

*Triacetyl* : m.p. 165°.

*Monobenzoyl* : prisms from AcOH or  $CHCl_3$ . M.p. 140°. Very sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .  $FeCl_3 \rightarrow$  green col.

*Dibenzoyl* : needles from toluene. M.p. 108°. Very sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ . No col. with  $FeCl_3$ .

*Tribenzoyl* : prisms from EtOH. M.p. 89-90°.

*Tri-phenylurethane* : needles from EtOH- $Et_2O$ . M.p. 173°. Very sol. EtOH. Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

*Tri-diphenylurethane* : m.p. 211.5-212.5°.

*Tricinnamoyl* : cryst. from EtOH. M.p. 136°.  $C_6H_6O_3 \cdot C_6H_5(NO_2)_3 \cdot 1 : 3 : 5$  : yellow needles. M.p. 163°.

*1-Me ether* :  $C_7H_8O_3$ . MW, 140. Needles. M.p. 38-41°. B.p. 129°/10 mm. *Diacetyl* : plates from EtOH. M.p. 91-3°. Spar. sol. EtOH. *Dibenzoyl* : cryst. from EtOH. M.p. 156-8°.

*2-Me ether* : cryst. from  $C_6H_6$ . M.p. 85-7°. B.p. 154-5°/24 mm. *Diacetyl* : plates from EtOH. M.p. 51-4°.

*1 : 2-Di-Me ether* :  $C_8H_{10}O_3$ . MW, 154. Clear oil. B.p. 233-4°, 124-5°/17 mm. *Benzoyl* : needles from pet. ether. M.p. 55-7°.

*1 : 3-Di-Me ether* : prisms from  $H_2O$ . M.p. 55-6°. B.p. 262-7°. Sol. 57 parts  $H_2O$  at 13°. *Acetyl* : cryst. from EtOH.Aq. M.p. 53.5°. *Benzoyl* : prisms. M.p. 118°.

*Tri-Me ether* :  $C_9H_{12}O_3$ . MW, 168. Needles from EtOH.Aq. M.p. 47°. B.p. 241°.  $D_4^{25}$  1.1118. Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .  $C_8H_{12}O_3 \cdot C_6H_5(NO_2)_3 \cdot 1 : 3 : 5$  : yellow prisms. M.p. 81°.

*Mono-Et ether* :  $C_8H_{10}O_3$ . MW, 154. Needles. M.p. 95° (102-4°). Very sol. EtOH,  $Et_2O$ . Spar. sol. cold  $H_2O$ ,  $C_6H_6$ .

*1 : 3-Di-Et ether* :  $C_{10}H_{14}O_3$ . MW, 182. Needles. M.p. 79-80°. B.p. 263-5°. Very sol.  $C_6H_6$ . Spar. sol. cold EtOH.Aq.

*Tri-Et ether* :  $C_{12}H_{18}O_3$ . MW, 210. Cryst. from EtOH.Aq. M.p. 39°. Insol. alkalis.

*Triallyl ether* : m.p. 3.5-4°.  $D_4^{20}$  1.04.  $n_D^{20}$  1.5265.

Marks, E.P., 140,694, (*Chem. Abstracts*, 1920, **14**, 2203).

Ullmann, *Ann.*, 1903, **327**, 116.

Einhorn, Hollandt, *Ann.*, 1898, **301**, 106.

Luynes, Esperandieu, *Compt. rend.*, 1865, **61**, 487.

### Pyrogallol-dicarboxylic Acid.

See 4 : 5 : 6-Trihydroxyisophthalic Acid and 3 : 4 : 5-Trihydroxyphthalic Acid.

### Pyroglutamic Acid.

See Glutimanic Acid.

**Pyroguaiacin** (6-Hydroxy-7-methoxy-2 : 3-dimethylnaphthalene)



$C_{13}H_{14}O_2$

MW, 202

Plates from EtOH. M.p. 183°. Mod. sol.  $Et_2O$ . Spar. sol. boiling  $H_2O$ , EtOH. Sublimes.

Zn dust. dist.  $\rightarrow$  2:3-dimethylnaphthalene.  $\text{H}_2\text{SO}_4 \rightarrow$  dark blue col. Gives cryst. Na and K salts.

*Acetyl*: needles from EtOH. M.p. 122–4°.

*Benzoyl*: cryst. from AcOH. M.p. 179°.

*6-Me ether*: 6:7-dimethoxy-2:3-dimethylnaphthalene.  $\text{C}_{14}\text{H}_{16}\text{O}_2$ . MW, 216. Plates from EtOH or formic acid, needles from ligroin. M.p. 149–50°. Spar. sol. EtOH, formic acid. Ox.  $\rightarrow$  6:7-dimethoxy-2:3-dimethyl-1:4-naphthoquinone. *Picrate*: red needles from MeOH. M.p. 130–1°.

Haworth, Mavin, *J. Chem. Soc.*, 1932, 1488.

Schroeter, Lichtenstadt, Irineu, *Ber.*, 1918, 51, 1604.

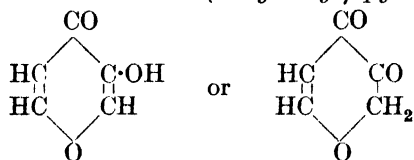
**Pyrohypaconitine.**

See under Hypaconitine.

**Pyroidone.**

See under Pyramidone.

**Pyromeonic Acid (3-Hydroxy- $\gamma$ -pyrone)**



$\text{C}_5\text{H}_4\text{O}_3$

MW, 112

Colourless prisms from  $\text{H}_2\text{O}$  or EtOH. M.p. 117°. B.p. 227–8°. Very sol.  $\text{H}_2\text{O}$ , EtOH. Sol.  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ . Possesses slight acid properties. Sublimes.  $\text{FeCl}_3 \rightarrow$  blood-red col. Forms unstable salts decomp. by  $\text{H}_2\text{O}$ . Does not combine with hydroxylamine. Gives cryst., spar. sol. ferric salt. Gives unstable hydrochloride. Red.  $\rightarrow$  3:4-dihydroxytetrahydropyran.

*Acetyl*: prisms from EtOH. M.p. 91°. Very sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{CHCl}_3$ . No col. with  $\text{FeCl}_3$ .

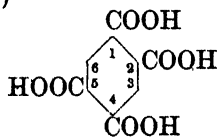
*Me ether*:  $\text{C}_6\text{H}_6\text{O}_2$ . MW, 126. Cryst. from  $\text{C}_6\text{H}_6$ -ligroin. M.p. 85°. Sol. boiling ligroin. Very sol. cold  $\text{H}_2\text{O}$ , hot  $\text{C}_6\text{H}_6$ ,  $\text{Et}_2\text{O}$ . Sublimes in vacuo.

*Et ether*:  $\text{C}_7\text{H}_8\text{O}_3$ . MW, 140. Colourless oil. B.p. 220–1°.

Borsche, *Ber.*, 1916, 49, 2544.

Ost, *J. prakt. Chem.*, 1879, 19, 183.

**Pyromellitic Acid (Benzene-1:2:4:5-tetracarboxylic acid)**



$\text{C}_{10}\text{H}_6\text{O}_8$

MW, 254

Prisms +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd. 275°. 1:4-*Di-Me ester*:  $\text{C}_{12}\text{H}_{10}\text{O}_8$ . MW, 282. M.p. 176–7°.

*Tetra-Me ester*:  $\text{C}_{14}\text{H}_{14}\text{O}_8$ . MW, 310. Leaflets from MeOH. M.p. 141.5°.

*Tetra-Et ester*:  $\text{C}_{18}\text{H}_{22}\text{O}_8$ . MW, 366. M.p. 54°.

*Di-anhydride*:  $\text{C}_{10}\text{H}_2\text{O}_6$ . MW, 218. Needles. M.p. 286°. Sol.  $\text{Me}_2\text{CO}$ , AcOEt. Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , ligroin. Sublimes.

Philippi, Thelen, *Organic Syntheses*, 1930, X, 90.

v. Braun, Lemke, *Ber.*, 1924, 57, 682.

de Diesbach, Guhl, *Helv. Chim. Acta*, 1927, 10, 448.

Schroeter, *Ber.*, 1924, 57, 2023.

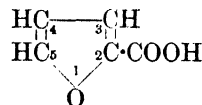
Fieser, Herschberg, *J. Am. Chem. Soc.*, 1935, 57, 1508.

I.G., D.R.P., 563,129, (*Chem. Abstracts*, 1933, 27, 1008).

Feist, *Ber.*, 1911, 44, 137.

Silberrad, *J. Chem. Soc.*, 1906, 89, 1795.

**Pyromucic Acid (Furan-2-carboxylic acid,  $\alpha$ -furoic acid)**



$\text{C}_5\text{H}_4\text{O}_3$

MW, 112

Leaflets from  $\text{H}_2\text{O}$ . M.p. 133–4°. B.p. 230–2°, 141–4°/20 mm. Part. sublimes at 100°, easily at 130–40°/50–60 mm. Sol. to 2.7% in  $\text{H}_2\text{O}$  at 0°. Sol. 28 parts  $\text{H}_2\text{O}$  at 15°, 4 parts at boil. Sol. EtOH,  $\text{Et}_2\text{O}$ .  $k = 7.6$  (7.07)  $\times 10^{-4}$  at 25°. Heat of comb.  $C_r$  494.4 Cal. At 275° under press. quantitatively  $\rightarrow$  furan +  $\text{CO}_2$ .  $\text{MnO}_2$  + conc. HCl  $\rightarrow$  mucochloric acid.

*Me ester*:  $\text{C}_6\text{H}_6\text{O}_3$ . MW, 126. B.p. 181.3°.  $D_4^{21.4}$  1.1786.  $n_D^{20}$  1.4860.

*Et ester*:  $\text{C}_7\text{H}_8\text{O}_3$ . MW, 140. Leaflets. M.p. 34°. B.p. 195°/766 mm., 128°/95 mm.  $D_4^{20.8}$  1.1174,  $D_4^{40}$  1.0974.  $n_D^{20.8}$  1.4797.

*Propyl ester*:  $\text{C}_8\text{H}_{10}\text{O}_3$ . MW, 154. B.p. 210.9°.  $D_4^{20.9}$  1.0745.  $n_D^{20.9}$  1.4737.

*Isopropyl ester*: b.p. 198.6°.  $D_4^{23.7}$  1.0655.  $n_D^{28.7}$  1.4682.

*Butyl ester*:  $\text{C}_9\text{H}_{12}\text{O}_3$ . MW, 168. B.p. 83–4°/1 mm.  $D_4^{20}$  1.0555.

*Isobutyl ester*: b.p. 220.8–222.6°.  $D_4^{23.5}$  1.0383.  $n_D^{27.5}$  1.4676.

*n-Amyl ester*:  $\text{C}_{10}\text{H}_{14}\text{O}_3$ . MW, 182. B.p. 95–7°/1 mm.  $D_4^{20}$  1.0335.

*n-Hexyl ester*:  $\text{C}_{11}\text{H}_{16}\text{O}_3$ . MW, 196. B.p. 105–7°/1 mm.  $D_4^{20}$  1.0170.



*n*-Heptyl ester:  $C_{12}H_{18}O_3$ . MW, 210. B.p.  $116-17^\circ/1$  mm.  $D_4^{20}$  1.0005.

Phenyl ester:  $C_{11}H_8O_3$ . MW, 188. Prisms from EtOH. M.p.  $42^\circ$ .

*p*-Nitrobenzyl ester: cryst. from 70% EtOH. M.p.  $133-5^\circ$ .

Anhydride:  $C_{10}H_6O_5$ . MW, 206. Needles from EtOH. M.p.  $73^\circ$ . B.p.  $325^\circ$  part. decomp.

Chloride:  $C_5H_3O_2Cl$ . MW, 130.5. M.p.  $-2^\circ$ . B.p.  $173^\circ$ ,  $66^\circ/10$  mm. Stable to  $H_2O$ .

Amide:  $C_5H_5O_2N$ . MW, 111. M.p.  $142-3^\circ$ . Part. sublimates at  $100^\circ$ .

Methylamide:  $C_6H_7O_2N$ . MW, 125. Cryst. from ligroin. M.p.  $64^\circ$ . B.p.  $250-3^\circ$ .

Ethylamide:  $C_7H_9O_2N$ . MW, 139. B.p.  $258^\circ$ .

Nitrile:  $C_5H_3ON$ . MW, 93. B.p.  $146^\circ/738$  mm.  $D_4^{20}$  1.0822.  $n_D^{20}$  1.4798.

Anilide: prisms from  $C_6H_6$ . M.p.  $123-5^\circ$ .

*o*-Toluidide: prisms from EtOH. M.p.  $62^\circ$ .

*m*-Toluidide: prisms from EtOH. M.p.  $87^\circ$ .

*p*-Toluidide: prisms from EtOH. M.p.  $107-5^\circ$ .

Wilson, *Organic Syntheses*, Collective Vol. I, 270.

Gennari, *Gazz. chim. ital.*, 1894, **24**, ii, 249.

Hughes, Johnson, *J. Am. Chem. Soc.*, 1931, **53**, 744.

Trickey, Miner, U.S.P., 1,665,236, (*Chem. Abstracts*, 1928, **22**, 1783).

Zanetti, Beckmann, *J. Am. Chem. Soc.*, 1926, **48**, 1068.

### Pyromucylacetic Acid.

See  $\alpha$ -Furoylacetic Acid.

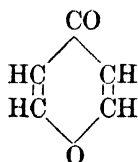
### Pyromucylformic Acid.

See  $\alpha$ -Furoylformic Acid.

### $\alpha$ -Pyrone.

See Coumalin.

### $\gamma$ -Pyrone



$C_5H_4O_2$  MW, 96

Hygroscopic cryst. M.p.  $32-5^\circ$ . B.p.  $215^\circ$ ,  $105^\circ/23$  mm.,  $97^\circ/13$  mm. Very sol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ ,  $AcOH$ . Sol.  $C_6H_6$ . Spar. sol. pet. ether,  $CS_2$ . Sol.  $KOH$ . Aq. with yellow col. Non-volatile in steam.  $NH_3$  at  $120-40^\circ \rightarrow$  4-hydroxypyridine.

$C_5H_4O_2 \cdot HCl$ : prisms from EtOH. M.p.  $139^\circ$ .

$C_5H_4O_2 \cdot (COOH)_2$ : plates. M.p.  $136-5^\circ$ .

$3C_5H_4O_2 \cdot (COOH)_2$ : prisms. M.p.  $139^\circ$ .

$3C_5H_4O_2 \cdot HAuCl_4$ : leaflets. M.p.  $116-5^\circ$ .

Picrate: needles from  $H_2O$  or EtOH. M.p.  $129^\circ$ .

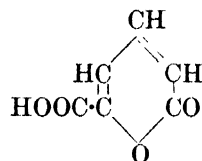
Willstätter, Pummerer, *Ber.*, 1904, **37**, 3745; 1905, **38**, 1465.

Ost, *J. prakt. Chem.*, 1884, **29**, 63.

### $\alpha$ -Pyrone-5-carboxylic Acid.

See Coumalic Acid.

### $\alpha$ -Pyrone-6-carboxylic Acid



$C_6H_4O_4$  MW, 140

Prisms from  $H_2O$ . M.p.  $228^\circ$ . Sublimes in needles. Spar. sol. EtOH,  $Me_2CO$ , hot  $H_2O$ . Insol.  $CHCl_3$ ,  $C_6H_6$ , pet. ether. Reduces  $NH_3 \cdot AgNO_3$ .

*Et ester*:  $C_8H_8O_4$ . MW, 168. Plates from ligroin. M.p.  $59-60^\circ$ . Sol.  $H_2O$  and most org. solvents.

Lapworth, *J. Chem. Soc.*, 1901, **79**, 1280.

Gault, *Compt. rend.*, 1914, **159**, 73.

### $\gamma$ -Pyrone-2-carboxylic Acid.

See Comanic Acid.

### $\gamma$ -Pyrone-2 : 6-dicarboxylic Acid.

See Chelidonic Acid.

### Pyroracemic Acid.

See Pyruvic Acid.

### Pyroracemic Alcohol.

See Hydroxyacetone.

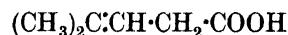
### Pyroracemic Aldehyde.

See Pyruvic Aldehyde.

### Pyrotartaric Acid.

See Methylsuccinic Acid.

**Pyroterebic Acid** (3-Methyl-2-butylene-1-carboxylic acid, 2-isopropylidene-propionic acid)



$C_6H_{10}O_2$  MW, 114

Oil with somewhat sharp odour. B.p.  $207-8^\circ$ ,  $111^\circ/22$  mm. Readily converted to isocapro-lactone.  $k = 2.51 \times 10^{-5}$  at  $25^\circ$ .

*Et ester*:  $C_8H_{14}O_2$ . MW, 142. B.p.  $58^\circ/11$  mm.  $D_4^{17}$  0.9134.  $n_D^{17}$  1.4329.

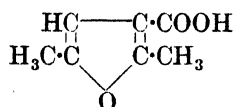
Nitrile:  $C_6H_7N$ . MW, 95. B.p.  $166^\circ$ ,  $65^\circ/20$  mm. Fuming  $HCl \rightarrow$  isocapro-lactone.

Anilide: needles from  $C_6H_6$ . M.p.  $106^\circ$ .

Fittig, Geisler, *Ann.*, 1881, **208**, 39.

Linstead, *J. Chem. Soc.*, 1929, 2506.

**Pyrotritaric Acid** (*Uvinic acid*, 2:5-dimethyl-furan-3-carboxylic acid, 2:5-dimethyl- $\beta$ -furoic acid)



$C_7H_8O_3$

MW, 140

Needles from hot  $H_2O$ . M.p.  $135^\circ$ . Sol. EtOH,  $Et_2O$ . Mod. sol. hot  $H_2O$ . Sublimes. Volatile in steam. Chromic mixture.  $\rightarrow CO_2 + CH_3COOH$ . Dil.  $HNO_3 \rightarrow CO_2 +$  oxalic acid.

*Me ester*:  $C_8H_{10}O_3$ . MW, 154. B.p.  $198^\circ$ .

*Et ester*:  $C_9H_{12}O_3$ . MW, 168. Oil. B.p.  $208-9^\circ$ ,  $99-101^\circ/14$  mm.,  $83-5^\circ/6$  mm.  $D_4^{23.1}$  1.0478.  $n_D^{23.1}$  1.4686.

*Hydrazide*: cryst. from  $H_2O$ . M.p.  $136^\circ$ .

Gilman, Burtner, *Rec. trav. chim.*, 1932, 51, 667.

Scott, Johnson, *J. Am. Chem. Soc.*, 1932, 54, 2555.

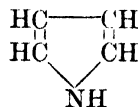
#### Pyrrocoline.

See Indolizine.

#### Pyrrodiazole.

See 1:2:4-Triazole.

#### Pyrrole



$C_4H_5N$

MW, 67

Present in coal tar. Colourless liq. with characteristic odour. B.p.  $130-1^\circ/761$  mm.  $D_4^{20}$  0.9691.  $n_D^{20}$  1.5085. Heat of comb.  $C_p$  567.5 Cal. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ . Turns brown in air. Forms metallic salts.  $CrO_3 + H_2SO_4 \rightarrow$  maleinimide. Polymerises with HCl to a trimer.

*N-Me*: see *N*-Methylpyrrole.

*N-Et*:  $C_6H_9N$ . MW, 95. Liq. with odour resembling pyrrole. B.p.  $131^\circ$ .  $D_4^{16}$  0.8881. Misc. with EtOH,  $Et_2O$ .

*N-Propyl*:  $C_7H_{11}N$ . MW, 109. B.p.  $146-5-147-5^\circ$ .  $D_4^{20}$  0.8833.

*N-Butyl*:  $C_8H_{13}N$ . MW, 123. B.p.  $165-80^\circ$ .

*N-Isoamyl*:  $C_9H_{15}N$ . MW, 137. B.p.  $180-4^\circ$ .  $D_4^{10}$  0.8786.

*N-Allyl*:  $C_7H_9N$ . MW, 107. B.p.  $105^\circ/48$  mm. Unstable in air. Volatile in steam.

*N-Phenyl*: see 1-Phenylpyrrole.

*N-o-Tolyl*: oil. B.p.  $246^\circ$ . Spar. sol. boiling  $H_2O$ . Volatile in steam.

Dict. of Org. Comp.—III.

*N-p-Tolyl*: leaflets from EtOH. M.p.  $82^\circ$ . B.p.  $252^\circ/728.5$  mm. Very sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ , pet. ether. Spar. sol. boiling  $H_2O$ . Volatile in steam.

*N-Benzyl*: b.p.  $245-6^\circ$ ,  $138-9^\circ/27$  mm. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ . Turns yellow in light and air.

*N-1-Naphthyl*: needles from EtOH.Aq. M.p.  $42^\circ$ . B.p. above  $360^\circ$ . Volatile in steam.

*N-2-Naphthyl*: cryst. from EtOH.Aq. M.p.  $107^\circ$ . B.p. above  $360^\circ$ . Volatile in steam.

*N-Formyl*: b.p.  $39^\circ/22$  mm.

*N-Acetyl*: b.p.  $181-2^\circ$ . Insol.  $H_2O$ .

*N-Propionyl*: yellowish oil. B.p.  $192-4^\circ$ .

*N-Benzoyl*: yellowish oil. B.p.  $276^\circ/715$  mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ , pet. ether. Insol.  $H_2O$ . Volatile in steam.

*Picrate*: orange-red cryst. M.p.  $69^\circ$  decomp.

McElvain, Bolliger, *Organic Syntheses*, Collective Vol. I, 461.

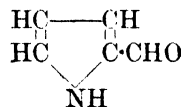
Pictet, *Ber.*, 1904, 37, 2792.

Jurjew, *Ber.*, 1936, 69, 440.

Jurjew, Schenjan, *Chem. Zentr.*, 1936, I, 4293.

Fischer, Orth, *Die Chemie des Pyrrols*, Vol. I (1934).

#### Pyrrole-2-aldehyde (2-Formylpyrrole)



$C_5H_5ON$

MW, 95

Prisms from pet. ether. M.p.  $46-7^\circ$ . B.p.  $217-19^\circ$ ,  $114^\circ/15$  mm.  $n_D^{15}$  1.5939. Heat of comb.  $C_p$  616.7 Cal. Volatile in steam. Does not oxidise in air.

*Oxime*: needles from  $CHCl_3$  or  $C_6H_6$ . M.p.  $164-5^\circ$ .

*Phenylhydrazones*: needles from ligroin. M.p.  $139-139.5^\circ$ .

*p-Nitrophenylhydrazones*: red needles from xylene. M.p.  $182.5-183^\circ$ .

*Semicarbazone*: leaflets from  $H_2O$ . M.p.  $183-5^\circ$ .

*N-Me*: see *N*-Methylpyrrole-2-aldehyde.

*N-Butyl*:  $C_9H_{13}ON$ . MW, 151. B.p. about  $75^\circ/2$  mm. *Semicarbazone*: cryst. from EtOH.Aq. M.p.  $146-147.5^\circ$ .

*N-Isoamyl*:  $C_{10}H_{15}ON$ . MW, 165. B.p. about  $90^\circ/2$  mm. *Semicarbazone*: cryst. from EtOH.Aq. M.p.  $155-7^\circ$ .

*N-Phenyl*: m.p.  $120^\circ$ . Decomp. in sunlight.

*N-Benzoyl*: cryst. from ligroin. M.p.  $90^\circ$ .

*Phenylhydrazone*: greenish-yellow plates from EtOH. M.p. 154°.

Emmert, Diehl, Gollwitzer, *Ber.*, 1929, **62**, 1737.

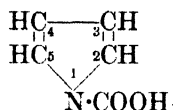
Putochin, *Ber.*, 1926, **59**, 1992.

Emmert, Diehl, *Ber.*, 1931, **64**, 131.

Reichstein, *Helv. Chim. Acta*, 1930, **13**, 352.

Alessandri, Passerini, *Gazz. chim. ital.*, 1921, **51**, 262.

**Pyrrole-N-carboxylic Acid** (*Pyrrole-1-carboxylic acid*)



$C_5H_5O_2N$  MW, 111

Prisms from Et<sub>2</sub>O. M.p. 95° decomp. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. At m.p. decomp. quantitatively into pyrrole + CO<sub>2</sub>. Resinifies with min. acids.

*Et ester*: C<sub>7</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 139. Liq. with characteristic odour. B.p. 180°/770 mm. Insol. H<sub>2</sub>O.

*Amide*: C<sub>5</sub>H<sub>6</sub>ON<sub>2</sub>. MW, 110. Leaflets from H<sub>2</sub>O. M.p. 166°. Sublimes.

Tschelinzeff, Maxoroff, *Ber.*, 1927, **60**, 196.

Ciamician, Magnaghi, *Ber.*, 1885, **18**, 416.

**Pyrrole-2-carboxylic Acid** (*Pyrrole-α-carboxylic acid*).

Leaflets from H<sub>2</sub>O. M.p. 208.5 (192°) decomp. → pyrrole + CO<sub>2</sub>. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. *k* (acid) = 4.0 × 10<sup>-5</sup> at 25°: *k* (base) = 3 × 10<sup>-13</sup>. Isoelectric point 2.9. The esters possess marked local anæsthetic properties.

*Me ester*: C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>N. MW, 125. Needles from pet. ether. M.p. 73°. B.p. 220-3°/740 mm., 120-30°/20-30 mm., 115-20°/12-16 mm. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, pet. ether.

*Et ester*: C<sub>7</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 139. Cryst. M.p. 39°. B.p. 230-2°. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, pet. ether. Very spar. sol. H<sub>2</sub>O.

*Propyl ester*: C<sub>8</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 153. B.p. 164-7°/50 mm.

*Butyl ester*: C<sub>9</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 167. M.p. 36-8°. B.p. 255-60°/740 mm.

*Isobutyl ester*: m.p. 68-9°. B.p. 250-5°/740 mm., 119-22°/70 mm.

*Isoamyl ester*: C<sub>10</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 181. B.p. 186-90°/100 mm.

*Chloride*: α-pyrroyl chloride. C<sub>5</sub>H<sub>4</sub>ONCl. MW, 127.5. Cryst. from Et<sub>2</sub>O-ligroin. Sinters

at 110°, decomp. at higher temps. Very sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Decomp. rapidly in moist air.

*Amide*: C<sub>5</sub>H<sub>6</sub>ON<sub>2</sub>. MW, 110. Plates from H<sub>2</sub>O or EtOH. M.p. 176°. Sweet taste.

*Anilide*: prisms from EtOH.Aq. M.p. 153-4°.

*N-Me*: C<sub>6</sub>H<sub>7</sub>O<sub>2</sub>N. MW, 125. M.p. 135°. *Methylamide*: prisms. M.p. 89-90°. Volatile in steam.

*N-Et*: C<sub>7</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 139. Needles from H<sub>2</sub>O. M.p. 78°. *Ethylamide*: prisms from H<sub>2</sub>O. M.p. 43-4°. B.p. 269-70°.

*N-Phenyl*: see 1-Phenylpyrrole-2-carboxylic Acid.

*Hydrazide*: cryst. from EtOH.Aq. M.p. 231-2° decomp.

*Azide*: cryst. M.p. 105° decomp.

Oddo, *Chem. Abstracts*, 1925, **19**, 2492.

Blicke, Blake, *J. Am. Chem. Soc.*, 1930, **52**, 238.

Schwanert, *Ann.*, 1860, **116**, 272.

Ciamician, Silber, *Ber.*, 1884, **17**, 104, 1152.

Bell, *Ber.*, 1877, **10**, 1866.

Fischer, van Slyke, *Ber.*, 1911, **44**, 3169.

Oddo, Moschini, *Gazz. chim. ital.*, 1912, **42**, ii, 254.

**Pyrrole-3-carboxylic Acid** (*Pyrrole-β-carboxylic acid*).

Needles. M.p. 161-2° decomp. Heat in vacuo → pyrrole + CO<sub>2</sub>.

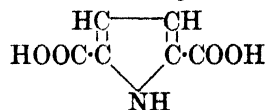
*Me ester*: needles or leaflets. M.p. 129°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. pet. ether.

Dennstedt, Zimmermann, *Ber.*, 1887, **20**, 855.

Ciamician, Silber, *Ber.*, 1884, **17**, 1438.

Oddo, Moschini, *Gazz. chim. ital.*, 1912, **42**, ii, 255.

**Pyrrole-2 : 5-dicarboxylic Acid**



$C_6H_5O_4N$  MW, 155

Needles from EtOH.Aq. Decomp. about 260°, part. → pyrrole + CO<sub>2</sub>. Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO. Insol. CHCl<sub>3</sub>, AcOEt, C<sub>6</sub>H<sub>6</sub>, pet. ether.

*Me ester*: C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>N. MW, 169. M.p. 243°.

*Di-Me ester*: C<sub>8</sub>H<sub>9</sub>O<sub>4</sub>N. MW, 183. Needles from H<sub>2</sub>O. M.p. 132°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Di-Et ester*: C<sub>10</sub>H<sub>13</sub>O<sub>4</sub>N. MW, 211. Needles. M.p. 82°.

*N-Me*: C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>N. MW, 169. Cryst. from H<sub>2</sub>O. Sinters and sublimes with part. decomp.

about 275°. *Di-Me'ester*:  $C_9H_{11}O_4N$ . MW, 197. Needles from EtOH.Aq. M.p. 80–1°.

*N-Et*:  $C_8H_9O_4N$ . MW, 183. Needles from EtOH.Aq. Decomp. at 250° without melting  $\rightarrow$  *N*-ethylpyrrole +  $CO_2$ . Insol.  $H_2O$ . *Diethylamide*: needles. M.p. 229–30°.

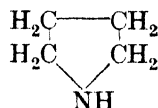
*N-Phenyl*:  $C_{12}H_9O_4N$ . MW, 231. Cryst. from  $H_2O$ . M.p. 235–40°  $\rightarrow$  *N*-phenylpyrrole +  $CO_2$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ . Insol. pet. ether.

Ciamician, Silber, *Ber.*, 1887, 20, 2595; 1886, 19, 1960.

Tschelinzew, Maxorow, *Chem. Zentr.*, 1923, I, 1507.

Bell, *Ber.*, 1877, 10, 1864.

**Pyrrolidine** (*Tetrahydropyrrole*, *tetramethyleineimine*)



$C_4H_9N$

MW, 71

Present in tobacco leaves. Liq. with odour resembling piperidine. B.p. 88.5–89°.  $D_{25}^{25}$  0.8520.  $n_D^{25}$  1.4270. Fumes in air. Misc. with  $H_2O$ . Strongly alkaline.

$B_2HAuCl_4$ : yellow cryst. from  $H_2O$ . M.p. 206° decomp.

$B_2, 2HI, CdI_2$ : leaflets or needles from  $H_2O$ . M.p. 217–19° (200–2°).

$B_2, H_2PtCl_6$ : orange prisms from EtOH.Aq. M.p. about 200° decomp.

*Picrate*: (i)  $B_2, C_6H_3O_7N_3$ : yellow needles from EtOH. M.p. 112°. (ii)  $B_2, C_6H_3O_7N_3$ : dark red cryst. M.p. 163–4°.

*N-Me*: see *N*-Methylpyrrolidine.

*N-Et*:  $C_6H_{13}N$ . MW, 99. B.p. 104°.  $D_4^{20}$  0.8156.  $n_D^{15}$  1.4113. *Picrate*: leaflets. M.p. 186°.

*N-Propyl*:  $C_7H_{15}N$ . MW, 113. B.p. 130°.  $D_4^{20}$  0.8171.  $n_D^{20}$  1.4389. Sol.  $H_2O$ . *Picrate*: m.p. 105° (101°).

*N-Butyl*:  $C_8H_{17}N$ . MW, 127. Slightly sol.  $H_2O$ . *Chloroaurate*: yellow leaflets from  $H_2O$ . M.p. 78°. *Picrate*: m.p. 124°.

*N-Amyl*:  $C_9H_{19}N$ . MW, 141. B.p. 179°, 89–94°/28 mm.  $D_4^{20}$  0.8216.  $n_D$  1.44276. *Chloroplatinate*: prisms from EtOH. M.p. 145°. *Picrate*: reddish-yellow needles from EtOH. M.p. 118–19°. *Methiodide*: m.p. 169–70°.

*N-Isoamyl*: b.p. 78°/32 mm.  $D_4^{20}$  0.8137.  $n_D$  1.43994. *Chloroaurate*: m.p. 158°.

*N-Allyl*:  $C_7H_{12}N$ . MW, 110. Volatile in steam and ether vapour. *Chloroaurate*: m.p. 97–8°. *Chloroplatinate*: m.p. 205°.

*N-Benzyl*:  $C_{11}H_{15}N$ . MW, 161. B.p. 237°. Sol. EtOH,  $Et_2O$ . Absorbs  $CO_2$  and  $H_2O$  from the air.  $B_2HAuCl_4$ : yellow prisms. M.p. 120° decomp.  $B_2, H_2PtCl_6$ : yellowish-red needles. M.p. 156° decomp. *Picrate*: m.p. 128°.

*N-Phenyl*: see 1-Phenylpyrrolidine.

*N-Benzoyl*: viscous liq. B.p. 190–1°/12 mm. Insol.  $H_2O$  and dil. acids. Dist. with  $PCl_5 \rightarrow$  benzonitrile + 1:4-dichlorobutane.

*N-Nitroso*: yellow oil. B.p. 214° decomp.

*N-p-Toluenesulphonyl*: cryst. from EtOH. M.p. 123°.

Schlinck, *Ber.*, 1899, 32, 951.

v. Braun, Beschke, *Ber.*, 1906, 39, 4121.

Ladenburg, *Ber.*, 1887, 20, 442.

v. Braun, *Ber.*, 1911, 44, 1252; 1916, 49, 2642.

de Jong, Wibaut, *Rec. trav. chim.*, 1930, 49, 237.

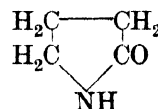
Yur'ev, Shen'yan, *Chem. Abstracts*, 1935, 29, 3335.

Ochiai, Tsuda, *Ber.*, 1934, 67, 1017.

**Pyrrolidine-2-carboxylic Acid.**

See Proline.

**2-Pyrrolidone** (*2-Ketopyrrolidine*, *butyrolactam*, *anhydride or lactam of piperidinic acid*)



$C_4H_7ON$

MW, 85

Cryst. from pet. ether. M.p. 24.6°. B.p. 245°, 133°/12 mm. (114°/14 mm.).  $D_4^{20}$  1.120,  $D_4^{40}$  1.097. Spar. volatile in steam. Aq. sol. reacts neutral to litmus. Very sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOEt,  $CS_2$ ,  $C_6H_6$ . Spar. sol. pet. ether. In moist air  $\rightarrow$  a monohydrate.

$B_2, H_2O$ : plates. M.p. 35° (29.7–29.9°).

$B_2, HCl$ : cryst. from  $Me_2CO$ . M.p. 86–8°.

$B_2, HCl$ : cryst. from  $Me_2CO$ . M.p. 128–31°.

$B_2, HBr$ : cryst. from  $CHCl_3$  or  $Me_2CO$ . M.p. 135–7°.

$B_2, HBr$ : cryst. M.p. 108–21°.

$B_2, H_2AuCl_4$ : yellow cryst. from  $H_2O$ . M.p. 82°.

*N-Me*: see *N*-Methyl-2-pyrrolidone.

*N-Et*:  $C_6H_{11}ON$ . MW, 113. Oil. B.p. 218°/751 mm.

*N-Isopropyl*:  $C_7H_{13}ON$ . MW, 127. Oil with peppermint odour. B.p. 221–2°/736 mm. Volatile.

*N-Phenyl*: see 1-Phenylpyrrolidone-2.

*N-p-Tolyl*: needles from  $H_2O$ . M.p. 88.5°. B.p. 189°/13 mm.

N-Acetyl: b.p. 231°/737 mm.

Tafel, Stern, *Ber.*, 1900, **33**, 2226.

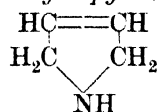
Gabriel, *Ber.*, 1889, **22**, 3338.

Späth, Breusch, *Monatsh.*, 1928, **50**, 356

### 2-Pyrrolidone-5-carboxylic Acid.

See Glutiminic Acid.

### 3-Pyrroline (Dihydropyrrole)



$\text{C}_4\text{H}_7\text{N}$

MW, 69

Liq. Fumes in air. B.p. 90–1°/750.5 mm.  $D_4^{20}$  0.9097.  $n_D^{20}$  1.4664. Sol.  $\text{H}_2\text{O}$ . Absorbs  $\text{CO}_2$  and  $\text{H}_2\text{O}$  from the air.

$B_2\text{HCl}$ : prisms from EtOH. M.p. 173–4°.

$B_2\text{HAuCl}_4$ : yellow prisms from  $\text{H}_2\text{O}$ . M.p. 152°.

$B_2\text{H}_2\text{PtCl}_6$ : orange-red cryst. from  $\text{H}_2\text{O}$ . M.p. 182°.

Picrolonate: yellow plates from EtOH. M.p. 260°.

N-Me: present in tobacco leaves. Oil with ammoniacal odour. B.p. 79–80°. Misc. with  $\text{H}_2\text{O}$ .  $B_2\text{HAuCl}_4$ : yellow leaflets from dil. HCl. M.p. 190–1°. Picrolonate: yellow prisms from  $\text{H}_2\text{O}$ . M.p. 222° decomp. Methiodide: cryst. from EtOH. M.p. 286°.

N-2:4-Dinitrophenyl: yellow needles from EtOH. M.p. 124–5°.

N-Benzyl: oil. B.p. about 150°.

N-Benzoyl: oil. B.p. 160–1°/2 mm.

N-Nitroso: needles from pet. ether. M.p. 37–8°.

Treibs, Dinelli, *Ann.*, 1935, **517**, 172.

Ciamician, Dennstedt, *Ber.*, 1883, **16**, 1536.

Knorr, Rabe, *Ber.*, 1901, **34**, 3497.

Dennstedt, D.R.P., 127,086, (*Chem. Zentr.*, 1902, I, 338).

### Pyrrone.

See 2:2'-Dipyreryl Ketone.

### Pyrrotriazole.

See 1:2:3:4-Tetrazole.

### 1-Pyrrylisopropyl Alcohol.

See 2-β-Hydroxypropylpyrrole.

**Pyruvic Acid** (1-Ketopropionic acid, pyruvic acid)



$\text{C}_3\text{H}_4\text{O}_3$

MW, 88

Liq. with odour resembling acetic acid. M.p. about 13.6°. B.p. 165° part. decomp., 75–80°/25 mm., 65°/10 mm.  $D_4^{15.3}$  1.2668.  $n_D^{15.3}$  1.43025. Misc. in all proportions with  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O.

Scratched with Pt wire  $\rightarrow$  a trimer, m.p. 92°. Warm conc.  $\text{HNO}_3 \rightarrow$  oxalic acid.  $\text{CrO}_3 \rightarrow$  acetic acid. Reduces  $\text{NH}_3\cdot\text{AgNO}_3$  and  $\text{HgCl}_2$ .  $k = 3.2 \times 10^{-3}$  at 25°.

Oxime: 1-isonitrosopropionic acid. Decomp. at 180–1° (177°).  $k = 5.14 (4.7) \times 10^{-4}$  at 25°. Acetate: cryst. M.p. 60° decomp.

Semicarbazone: needles from  $\text{H}_2\text{O}$ . M.p. about 200° decomp.

Phenylhydrazone: needles from  $\text{H}_2\text{O}$ . M.p. 192°.

o-Nitrophenylhydrazone: yellow needles from EtOH. M.p. 221°.

p-Nitrophenylhydrazone: yellow cryst. from EtOH. M.p. 220°.

2:4-Dinitrophenylhydrazone: yellow cryst. from AcOH. M.p. 218° (213°).

p-Bromophenylhydrazone: yellow needles from AcOH. M.p. 184°.

m-Nitrobenzoylhydrazone: m.p. 185.5–186.5°.

Me ester:  $\text{C}_4\text{H}_6\text{O}_3$ . MW, 102. B.p. 134–7°.

$D^0$  1.154. Oxime: needles from Et<sub>2</sub>O. M.p.

69°. B.p. 122–3°/14 mm. Oxime acetate: m.p.

42°. B.p. 126°/14 mm. 2:4-Dinitrophenyl-

hydrazone: m.p. 186.5–187.5°. Di-Me acetal:

b.p. 66–66.5°/16 mm.  $D_4^{17.6}$  1.0678.  $n_D^{17.6}$  1.412.

Et ester:  $\text{C}_5\text{H}_8\text{O}_3$ . MW, 116. B.p. 155°

(144°), 55°/17 mm.  $D_4^{15.6}$  1.0596.  $n_D^{15.6}$  1.408.

Oxime: prisms or needles. M.p. 97°. B.p.

213° slight decomp. Semicarbazone: m.p.

206° decomp. Phenylhydrazone: two forms.

(i) M.p. 117–18°. (ii) M.p. 31–2°. p-Nitro-

phenylhydrazone: yellow cryst. from EtOH.

M.p. 187°. 2:4-Dinitrophenylhydrazone: yel-

low cryst. from dioxan-EtOH. M.p. 154.5–155°.

Di-Et acetal: b.p. 190–1° slight decomp., 81.5–

82.5°/15 mm.  $D_4^{18.2}$  0.9783.  $n_D^{17.1}$  1.415.

Propyl ester:  $\text{C}_6\text{H}_{10}\text{O}_3$ . MW, 130. B.p.

166°. Semicarbazone: m.p. 178°. 2:4-Dinitro-

phenylhydrazone: m.p. 119–20°.

d-Amyl ester:  $\text{C}_8\text{H}_{14}\text{O}_3$ . MW, 158. B.p.

185–6°. 85–6°/16 mm., 81–2°/10 mm.  $D_4^{13}$

0.9724.  $n_D^{18}$  1.4206.  $[\alpha]_D^{15} + 3.25^\circ$ .

Isoamyl ester: b.p. 185°, 86°/14 mm.  $D^{17}$

0.978.

Hexadecyl ester:  $\text{C}_{19}\text{H}_{36}\text{O}_3$ . MW, 312. M.p.

26.5–27.5°. Semicarbazone: m.p. 140–1°.

Allyl ester:  $\text{C}_6\text{H}_8\text{O}_3$ . MW, 128. B.p. 165°,

65°/14 mm.  $D_4^{17.6}$  1.082.

1-Menthyl ester: oil. B.p. 136–40°/22 mm.,

131–2°/10 mm.  $D_4^{19.6}$  0.9852.  $[\alpha]_D^{20} - 82.00^\circ$  in

$\text{CHCl}_3$ .

Benzyl ester: b.p. 207–8°, 103–4°/36 mm.  $D^{14}$

1.090. Semicarbazone: m.p. 176°.

Amide:  $\text{C}_3\text{H}_5\text{O}_2\text{N}$ . MW, 87. Prisms or

plates from EtOH. M.p. 124–5°. Sol.  $\text{H}_2\text{O}$ ,

$\text{CHCl}_3$ , hot  $\text{C}_6\text{H}_6$ . *Oxime*: plates or prisms from  $\text{H}_2\text{O}$ . M.p. 178.5° decomp. *Semicarbazone*: m.p. 230° decomp.

*Nitrile*: acetyl cyanide.  $\text{C}_3\text{H}_3\text{ON}$ . MW, 69. Liq. with characteristic odour. B.p. 93°.  $D_4^{20}$  0.9745.  $n_D^{20}$  1.3743. *Phenylhydrazones*: leaflets from  $\text{C}_6\text{H}_6$ . M.p. 150–1°. *Semicarbazone*: m.p. 215° decomp.

*Anil*: 1-phenyliminopropionic acid, anil-pyruvic acid. M.p. 127–8° decomp. Sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol. AcOEt,  $\text{C}_6\text{H}_6$ . Very spar. sol.  $\text{CHCl}_3$ . Sol. conc.  $\text{H}_2\text{SO}_4$  with wine-red col. *o-Tolylimide*: m.p. 146° decomp.

*p-Tolylimide*: m.p. 127°.

*Anilide*: needles from  $\text{H}_2\text{O}$  or EtOH. M.p. 104°. Sublimes. *Oxime*: yellowish plates from EtOH. M.p. 119°.

*N-Me-anilide*: cryst. from  $\text{H}_2\text{O}$ . M.p. 152–3°.

*o-Toluidide*: needles from EtOH. M.p. 70–1°.

*p-Toluidide*: plates from  $\text{C}_6\text{H}_6$ . M.p. 109°.

*Oxime*: leaflets from EtOH. M.p. 130°.

Howard, Fraser, *Organic Syntheses*, Collective Vol. I, 462.

Fernbach, Strange, E.P., 14,607, (*Chem. Abstracts*, 1919, 13, 1595).

Boehringer Sohn, D.R.P., 523,190, (*Chem. Zentr.*, 1931, II, 496).

Tschelinzeff, Schmidt, *Ber.*, 1929, 62, 2211.

### Pyruvic Alcohol.

See Hydroxyacetone.

**Pyruvic Aldehyde** (1-Ketopropionaldehyde, methylglyoxal, pyroracemic aldehyde, propanalone)



$\text{C}_3\text{H}_4\text{O}_2$

MW, 72

Yellow liq. with pungent odour. Begins to boil at 72° → a yellowish-green vapour. Liq. at room temp. is bimolecular, rapidly polymerising to an amorphous glassy mass of unknown MW. Latter at 50° → bimolecular liq. or monomolecular vap.

*Oxime*: see Isonitrosoacetone.

*Dioxime*: see Methylglyoxime.

*Phenylosazone*: m.p. 145° (154.8°).

*Disemicarbazone*: m.p. 254°.

*o-Nitrophenylhydrazones*: yellow needles from EtOH. M.p. 128°.

*m-Nitrophenylhydrazones*: pale yellow needles from EtOH. M.p. 152°.

*p-Nitrophenylhydrazones*: yellow needles from EtOH. M.p. 217°.

*Di-p-nitrophenylhydrazones*: m.p. 302–4°.

*Di-2:4-dinitrophenylhydrazones*: reddish-orange cryst. from  $\text{PhNO}_2$ . M.p. 299–300°.

*Di-m-nitrobenzoylhydrazones*: m.p. 288.5°.

*Dihydrazones*: cryst. from EtOH. M.p. 93–4°.

*Di-Me acetal*: b.p. 143–7°.

*Di-Et acetal*: liq. with odour resembling acetone. B.p. 161.7–161.8°/761 mm., 54–5°/13–15 mm.

*Tetra-Et acetal*: b.p. 192°.

*Diacetate*: pale yellow liq. B.p. 115–16°/13 mm.

Meisenheimer, *Ber.*, 1912, 45, 2637.

Neuberg, Färber, Levite, Schwenk, *Biochem. Z.*, 1917, 83, 264.

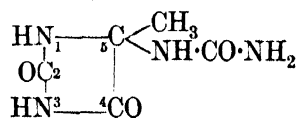
Dakin, Dudley, *J. Biol. Chem.*, 1913, 15, 130.

Neuberg, Dalmer, *Biochem. Z.*, 1925, 162, 488.

Fischer, Taube, *Ber.*, 1924, 57, 1506.

Henze, Müller, *Z. physiol. Chem.*, 1933, 214, 281.

### Pyruvil (5-Methylallantoin)



$\text{C}_5\text{H}_8\text{O}_3\text{N}_4$

MW, 172

Plates from  $\text{H}_2\text{O}$ . Insol. EtOH,  $\text{Et}_2\text{O}$ . Heat of comb. 567.7 Cal.

Davidson, *J. Am. Chem. Soc.*, 1925, 47, 255.

Simon, *Compt. rend.*, 1901, 133, 587.

### Pyruvylformic Acid.

See Diketobutyric Acid.

## Q

**p-Quaterphenyl.**Benzerythrene, *q.v.***Quebrachamine** $C_{19}H_{26}N_2$ 

MW, 282

Isolated from quebracho bark. Leaflets from EtOH or  $C_6H_6$ . M.p.  $147^\circ$ .  $[\alpha]_D -109.5^\circ$  in  $Me_2CO$ . Weak tertiary base. Sol.  $Me_2CO$ . Mod. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

$B, H_2SO_4, 2H_2O$ : prisms. Spar. sol. cold  $H_2O$ .

$B, (COOH)_2$ : prisms from EtOH. M.p.  $217^\circ$ .

*Picrate*: scarlet needles from EtOH. M.p.  $195-6^\circ$ .

*Methiodide*: cream-coloured prisms from MeOH. M.p.  $234^\circ$ . Sol. EtOH,  $Me_2CO$ . Insol.  $Et_2O$ ,  $C_6H_6$ .

*Methosulphate*: prisms. M.p.  $235^\circ$ . Sol. EtOH. Mod. sol.  $H_2O$ .

Hesse, *Ann.*, 1882, 211, 249.

Field, *J. Chem. Soc.*, 1924, 125, 1444.

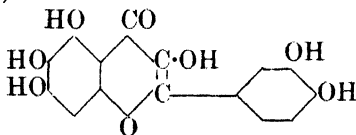
**Quebrachine.**

See Yohimbine.

**Quebrachitol.**

See under Inositol.

**Quercetagenin** (3 : 5 : 6 : 7 : 3' : 4'-Hexahydroxyflavone)

 $C_{15}H_{10}O_8$ 

MW, 318

Isolated from flowers of African marigold (*Tagetes patula*). Cryst.  $+2H_2O$  from dil. EtOH. Pale yellow needles or leaflets. M.p.  $318-20^\circ$ . Sol. hot EtOH, dil. alkalis. Spar. sol. boiling  $H_2O$ . Ox. by air becoming olive-green and finally brown.  $FeCl_3$  in EtOH  $\rightarrow$  olive-green col. Lead acetate in EtOH  $\rightarrow$  orange-red ppt. KOH fusion  $\rightarrow$  protocatechuic acid. Forms oxonium salts with min. acids.

$C_{15}H_{10}O_8, H_2SO_4$ : orange needles. Hyd. by  $H_2O$ .

*Hexacetyl*: needles from EtOH-AcOH. M.p.  $209-11^\circ$  ( $203-5^\circ$ ).

*Penta-Me ether*: pale yellow needles from EtOH. M.p.  $161-2^\circ$ . *Acetyl*: m.p.  $161-3^\circ$ .

*Hexa-Me ether*: colourless needles from  $Me_2CO$ . M.p.  $157-8^\circ$ .

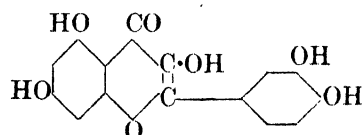
*Hexa-Et ether*: cryst. from EtOH. M.p.  $139-41^\circ$ .

Latour, Magnier de la Source, *Bull. soc. chim.*, 1877, 28, 337.

Perkin, *J. Chem. Soc.*, 1913, 103, 209.

Baker, Nodzu, Robinson, *J. Chem. Soc.*, 1929, 74.

**Quercetin** (*Meletin, sophoretin, quercitin, 3 : 5 : 7 : 3' : 4'-pentahydroxyflavone*)

 $C_{15}H_{10}O_7$ 

MW, 302

Found in rind of many fruits. Yellow needles  $+2H_2O$  from EtOH.Aq. M.p. anhyd.  $313-14^\circ$ . Very sol. aq. alkalis with golden-yellow col. Sol. AcOH, boiling EtOH. Spar. sol. hot  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  yellow sol. with faint green fluor. Reduces  $NH_3, AgNO_3$  in cold and Fehling's on heating. Tasteless.

*7-Me ether*: see Rhamnetin.

*3'-Me ether*: see Isorhamnetin.

*7 : 3'-Di-Me ether*: see Rhamnazin.

*5-Me ether*:  $C_{16}H_{12}O_7$ . MW, 316. Prismatic needles from EtOH. M.p.  $305-8^\circ$ . Alkalis  $\rightarrow$  orange sols. *Tetra-acetyl*: needles from  $Me_2CO$ . M.p.  $202-4^\circ$ .

*3 : 3' : 4'-Tri-Me ether*:  $C_{18}H_{16}O_7$ . MW, 344. Pale yellow needles from AcOEt. M.p.  $240-5^\circ$ . Alc.  $FeCl_3 \rightarrow$  intense greenish-brown col. Conc.  $H_2SO_4 \rightarrow$  yellow sol. with weak green fluor. *Diacetyl*: needles from EtOH. M.p.  $159-60^\circ$ .

*3 : 5 : 3' : 4'-Tetra-Me ether*:  $C_{19}H_{18}O_7$ . MW, 358. Needles from EtOH. M.p.  $284-5^\circ$ . Spar. sol. EtOH. Alkalis  $\rightarrow$  pale yellow sols. *Acetyl*: needles. M.p.  $174-6^\circ$ .

*3 : 7 : 3' : 4'-Tetra-Me ether*: pale yellow needles from EtOH. M.p.  $159-60^\circ$  ( $155^\circ$ ). Spar. sol. EtOH. Alc. KOH  $\rightarrow$  bright yellow col. *Acetyl*: needles from EtOH. M.p.  $169-70^\circ$ . Spar. sol. cold EtOH.

*5 : 7 : 3' : 4'-Tetra-Me ether*: needles from EtOH. M.p.  $197-8^\circ$ . Conc.  $H_2SO_4 \rightarrow$  yellow sol. with green fluor. *Acetyl*: needles from EtOH. M.p.  $160-3^\circ$ .

*Penta-Me ether*:  $C_{20}H_{20}O_7$ . MW, 372.

Needles from EtOH or AcOEt. M.p. 151–2°. Gives no col. with alc. KOH.

7 : 3' : 4'-*Tri-Et ether* :  $C_{21}H_{22}O_7$ . MW, 386. Yellow needles from EtOH. M.p. 123–4°.

3 : 7 : 3' : 4'-*Tetra-Et ether* :  $C_{23}H_{26}O_7$ . MW, 414. Yellow needles. M.p. 121–2°. Mod. sol. cold EtOH. Insol.  $H_2O$ . *Acetyl* : needles from 70% EtOH. M.p. 152–3°. Mod. sol. cold EtOH. Dil. alc. sol. shows faint blue fluor.

*Penta-Et ether* :  $C_{25}H_{30}O_7$ . MW, 442. Needles M.p. 116–18°.

3 : 7 : 3' : 4'-*Tetra-acetyl* : needles. M.p. 193–4°.

*Penta-acetyl* : needles from EtOH or  $C_6H_6$ . M.p. 193.5° (190–1°).

*Pentachloroacetyl* : needles. M.p. 180°.

*Pentabenzoyl* : needles from hot  $Me_2CO$ . M.p. 188–90°.

3-*Glucoside* : see Isoquercitrin.

7-*Glucoside* : see Quercimeritrin.

3-*Rhamnoside* : see Quercitrin.

3-*Rutinoside* : see Rutin.

?-*Glucoside* : incarnatrin.  $C_{21}H_{20}O_{12}$ . MW, 464. In flowers of *Trifolium incarnatum*, Linn. Yellow needles +  $3H_2O$  from  $H_2O$ . Softens at 165°, decomp. at 242–5°. Position of glucose not known.

Perkin, Pate, *J. Chem. Soc.*, 1895, **67**, 646.

v. Kostanecki, Lampe, Tambor, *Ber.*, 1904, **37**, 1404.

Herzig, *Monatsh.*, 1888, **9**, 541; 1912, **33**, 690.

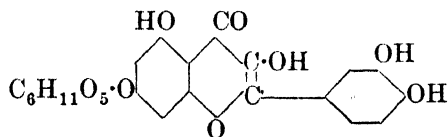
Attree, Perkin, *J. Chem. Soc.*, 1927, 239.

Allan, Robinson, *J. Chem. Soc.*, 1926, 2336.

Kubota, Perkin, *J. Chem. Soc.*, 1925, 1894.

Rogerson, *J. Chem. Soc.*, 1910, **97**, 1004.

### Quercimeritrin (Quercetin 7-glucoside)



$C_{21}H_{20}O_{12}$

MW, 464

Isolated from cotton flowers, leaves of *Gossypium hirsutum*, *Helianthus annuus*, and from bark of *Prunus seratina*, Ehrh. Yellow plates. M.p. 247–9°. Yellow needles +  $3H_2O$  from Aq.Py. M.p. 246–8°. Loses  $H_2O$  at 100°. Prac. insol. hot  $H_2O$ . Hyd.  $\rightarrow$  quercetin + glucose.

*Octa-acetyl* : m.p. 216–17° (214–16°).

*Penta-Me ether* : cryst. +  $2H_2O$ . Sinters at 197°. M.p. 203–5°.

Perkin, *J. Chem. Soc.*, 1909, **96**, 2185.

Sando, *J. Biol. Chem.*, 1925, **64**, 71; 1926, **68**, 407.

Attree, Perkin, *J. Chem. Soc.*, 1927, 237.

Neelakantan, Rao, Seshadri, *Chem. Zentr.*, 1936, I, 3518.

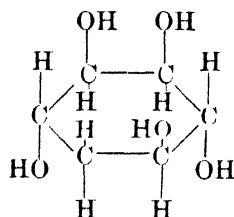
### Quercin.

See Scyllitol.

### Quercitin.

See Quercetin.

### d-Quercitol (Pentahydroxycyclohexane)



$C_6H_{12}O_5$

MW, 164

Occurs in acorns, oak bark, leaves of *Chamaerops humilis*, Linn., seeds of *Mimusops elengi*, and *Achras sapota*, Linn. Prisms from  $H_2O$  or dil. EtOH. M.p. 234° (232°, 235–7°). Sol.  $H_2O$ . Insol. cold EtOH,  $Et_2O$ .  $[\alpha]_D^{15} + 24.37^\circ$  in  $H_2O$ ,  $[\alpha]_D^{20} + 25.6^\circ$  in  $H_2O$ . Non-fermentable.  $HNO_3 \rightarrow$  mucic acid.  $KMnO_4 \rightarrow$  oxalic and malonic acids. d- and l-quercitol are not optical antipodes.

*Monoacetyl* : cryst. Sol.  $Et_2O$ .

*Diacetyl* : hard friable mass. Sol. EtOH.

*Triacetyl* : amorph. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

*Tetra-acetyl* : amorph. brittle mass. Hygroscopic.

*Penta-acetyl* : amorph. Sol.  $Et_2O$ . Mod. sol. EtOH. Spar. sol.  $H_2O$ .

*Pentacarbanilate* : amorph. M.p. 120–40°. Insol. ligroin.

Prunier, *Ann. chim. phys.*, 1878, **15**, 9.

Kiliani, Schäfer, *Ber.*, 1896, **29**, 1762.

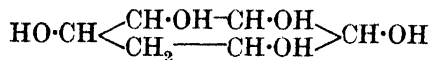
Missenden, *Chem. News*, 1922, **125**, 120.

Karrer, *Helv. Chim. Acta*, 1926, **9**, 116.

Kiliani, *Ber.*, 1931, **64**, 2473.

Posternak, *Helv. Chim. Acta*, 1932, **15**, 948.

### l-Quercitol (Pentahydroxycyclohexane)



$C_6H_{12}O_5$

MW, 164



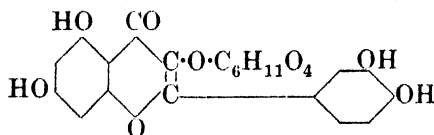
Occurs in leaves of *Gymnema sylvestre*, R.Br. Prisms +  $H_2O$  from  $H_2O$  or needles from EtOH. M.p.  $174^\circ$ .  $[\alpha]_D^{20} - 73.9^\circ$  in  $H_2O$ . Sol.  $H_2O$ . Prac. insol. EtOH.

*Penta-acetyl*: needles from dil. EtOH, m.p.  $124-5^\circ$ : cryst. +  $C_6H_6$  from  $C_6H_6$ , m.p.  $87-97^\circ$ .  $[\alpha]_D - 26.0^\circ$  in  $CHCl_3$ .

*Pentabenzoyl*: amorph. from EtOH, m.p.  $133^\circ$ : needles + EtOH from pet. ether-AcOEt-EtOH, m.p.  $116^\circ$ .  $[\alpha]_D - 79^\circ$  in  $CHCl_3$ .

Power, Tutin, *J. Chem. Soc.*, 1904, 85, 624.

### Quercitrin (*Quercetin 3-rhamnoside*)



$C_{21}H_{20}O_{11}$

MW, 448

Occurs in quercitron bark. Pale yellow leaflets +  $2H_2O$ . M.p.  $182-5^\circ$  (air dried),  $250-2^\circ$  anhyd. Sol. EtOH. Insol. cold  $H_2O$ . Gives yellow ppt. with aq. lead acetate. Hyd.  $\rightarrow$  quercetin + rhamnose.

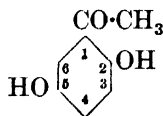
*Hepta-acetyl*: powder.  $[\alpha]_D^{24} - 165.6^\circ$  in  $CHCl_3$ . Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ ,  $Et_2O$ , pet. ether.

Zwenger, Dronke, *Ann., Suppl.*, 1861, 1, 267.

Liebermann, Hamburger, *Ber.*, 1879, 12, 1179.

Zemplén, Csürös, Gerecs, Aczél, *Ber.*, 1928, 61, 2486.

### Quinacetophenone (*Acetohydroquinone*, 2:5-dihydroxyacetophenone)



$C_8H_8O_3$

MW, 152

Yellowish-green cryst. from  $H_2O$ . M.p.  $202^\circ$ . Sol. EtOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Prac. insol. cold  $H_2O$ . Sol. alkalis with yellow col.  $FeCl_3 \rightarrow$  transient blue col. Sublimes.

5-*Me ether*:  $C_9H_{10}O_3$ . MW, 166. Yellow prisms from dil. EtOH. M.p.  $52^\circ$ .

2:5-*Di-Me ether*:  $C_{10}H_{12}O_3$ . MW, 180. Cryst. M.p.  $20-2^\circ$ . B.p.  $155-8^\circ/14$  mm.  $D_4^{20} 1.1385$ . *Semicarbazone*: needles from dil. EtOH. M.p.  $181-2^\circ$ . *Phenylhydrazone*: yellow prisms from EtOH. M.p.  $99-100^\circ$ .

5-*Et ether*:  $C_{10}H_{12}O_3$ . MW, 180. Yellow prisms from EtOH. M.p.  $57^\circ$ .

2:5-*Di-Et ether*:  $C_{12}H_{16}O_3$ . MW, 208. Cryst. from EtOH. M.p.  $42^\circ$ .

5(?)*-Acetyl*: yellow needles from AcOH. M.p.  $91^\circ$ . *Phenylhydrazone*: needles from dil. EtOH or  $C_6H_6$ . M.p.  $147^\circ$ .

2:5-*Diacetyl*: needles from AcOH. M.p.  $68^\circ$ .

2:5-*Dibenzoyl*: prisms from EtOH- $C_6H_6$ . M.p.  $113^\circ$ . *Phenylhydrazone*: yellow needles from dil. EtOH. M.p.  $148^\circ$ .

*Oxime*: plates from toluene. M.p.  $149-50^\circ$ .

Nencki, Schmid, *J. prakt. Chem.*, 1881, 23, 546.

Klinger, Kolvenbach, *Ber.*, 1898, 31, 1214.

Kostanecki, Lampe, *Ber.*, 1904, 37, 774 (Footnote).

Kauffmann, Beisswenger, *Ber.*, 1905, 38, 791.

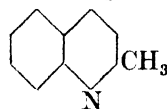
### Quinaform.

See under Quinine.

### Quinaldic Acid.

See Quinaldinic Acid.

### Quinaldine (2-Methylquinoline)



$C_{10}H_9N$

MW, 143

Present in coal tar. F.p.  $-2^\circ$  to  $-1^\circ$ . B.p.  $247.6^\circ/760$  mm.,  $238-9^\circ/716$  mm.,  $135.5^\circ/26$  mm.,  $118^\circ/10$  mm.  $D_4^{20} 1.0585$ .  $n_D^{20} 1.6126$ . Heat of comb.  $C_7$  1286.27 Cal.  $k = 3.6 \times 10^{-9}$  at  $14^\circ$ ,  $7.4 \times 10^{-9}$  at  $25^\circ$ .  $CrO_3 \rightarrow$  quinaldinic acid.  $KMnO_4 \rightarrow$  acetylanthranilic acid.  $Sn + HCl \rightarrow$  1:2:3:4-tetrahydroquinaldine. Conc.  $HNO_3 \rightarrow$  nitroquinaldinic acid.  $HNO_3 + H_2SO_4 \rightarrow$  5- and 8-nitroquinaldines.  $H_2SO_4 \rightarrow$  quinaldine-5-, -6-, and -8-sulphonic acids.  $SeO_2 \rightarrow$  quinoline-2-aldehyde and quinaldinic acid.

$B, HCl$ : needles from EtOH- $Et_2O$ . M.p.  $224^\circ$ .

$B, 2HCl$ : m.p. about  $31^\circ$ .

$B, HBr$ : cryst. +  $3H_2O$ . M.p.  $54^\circ$ .

$B, HI$ : needles from EtOH. M.p.  $186^\circ$ . Sol.  $H_2O$ .

$B, H_2SO_4$ : prisms. M.p.  $211-13^\circ$ .

$B, H_2PtCl_6$ : orange-red prisms from  $H_2O$ . M.p.  $228-9^\circ$  ( $226^\circ$ ,  $230-41^\circ$ ). Less sol. hot  $H_2O$  than quinoline chloroplatinate.

$B, H_2Cr_2O_7$ : yellowish-red needles. Sol. hot  $H_2O$ . Also yellow needles +  $3H_2O$  for  $H_2O$ . M.p.  $110^\circ$ .

$B, 2HCl, ZnCl_2$ : decomp. at  $245^\circ$ .

$B, HCl, HgCl_2$ : needles. M.p.  $165.5^\circ$ .

$B, 2HCl, CuCl_2$ : orange tablets. M.p.  $175-8^\circ$  decomp.

*Picrate*: yellow needles. M.p. 194° (191°). Spar. sol. H<sub>2</sub>O, cold EtOH.

*Styphnate*: m.p. 213–14°.

*N-Oxide*: needles + H<sub>2</sub>O (or ½H<sub>2</sub>O) from H<sub>2</sub>O. M.p. 77–8°. Decomp. at 200°. Sol. most org. solvents and acids. Insol. alkalis. *B<sub>4</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow prisms + 2H<sub>2</sub>O. M.p. 207° decomp. *Picrate*: yellow cryst. from EtOH. M.p. about 173°.

*Methiodide*: yellow needles from EtOH. M.p. 195°. Sol. H<sub>2</sub>O. Insol. Et<sub>2</sub>O.

*Ethiodide*: yellow needles from EtOH. M.p. 233–4°. Sol. H<sub>2</sub>O. Spar. sol. EtOH. Insol. Et<sub>2</sub>O.

*Propiodide*: greenish-yellow prisms from EtOH. M.p. 166–7° decomp.

*Isobutylidide*: yellow plates from EtOH. M.p. 172°.

*Isoamylidide*: yellow prisms from EtOH. M.p. 175°. Sol. H<sub>2</sub>O. Spar. sol. cold EtOH.

*Methyl-p-toluenesulphonate*: cryst. M.p. 134°.

*Ethyl-benzenesulphonate*: cryst. M.p. 105°.

*Methoperchlorate*: prisms from MeOH. M.p. 154°. Mod. sol. cold H<sub>2</sub>O.

Doebner, v. Miller, *Ber.*, 1883, **16**, 2465.

Skraup, *Ber.*, 1882, **15**, 897.

Schering, D.R.P., 24,317.

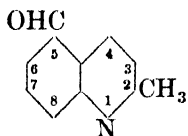
Mills, Harris, Lambourne, *J. Chem. Soc.*, 1921, **119**, 1297.

Basu, *Ann.*, 1934, **512**, 134.

Rabinovich, Dzirkal, *Chem. Abstracts*, 1934, **28**, 3725.

Tseou Heou-Feo, *Bull. soc. chim.*, 1935, **2**, 90.

### Quinaldine-5-aldehyde (2-Methylquinoline-5-aldehyde, 5-aldehydoquinaldine)



C<sub>11</sub>H<sub>9</sub>ON

MW, 171

Cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 73°, anhyd. 61°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, dil. min. acids. Spar. sol. hot H<sub>2</sub>O, pet. ether. Volatile in steam. Ag<sub>2</sub>O → quinaldine-5-carboxylic acid.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-yellow plates from alc. HCl. M.p. 211°. Spar. sol. hot EtOH.

*Picrate*: needles from EtOH. M.p. 182° decomp.

Eckhardt, *Ber.*, 1889, **22**, 277.

See also Decker, Remfry, *Ber.*, 1905, **38**, 2775.

### Quinaldine-6-aldehyde (2-Methylquinoline-6-aldehyde, 6-aldehydoquinaldine).

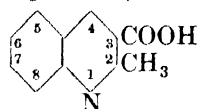
Needles from H<sub>2</sub>O, plates from C<sub>6</sub>H<sub>6</sub>-pet. ether, m.p. 106°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, min. acids. Mod. sol. hot H<sub>2</sub>O, pet. ether.

*Phenylhydrazone*: golden-yellow prisms from EtOH. M.p. 160°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange prisms + 2H<sub>2</sub>O.

v. Miller, Kinkelin, *Ber.*, 1885, **18**, 3237.

### Quinaldine-3-carboxylic Acid (2-Methylquinoline-3-carboxylic acid)



C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 187

Needles from EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 238° (235° decomp., 251° decomp.). Spar. sol. most solvents. Prac. insol. H<sub>2</sub>O. Heat → quinaldine.

*Me ester*: C<sub>12</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 201. Needles from EtOH. M.p. 72°. *Methiodide*: yellow needles or plates from H<sub>2</sub>O. M.p. 200° decomp. Sol. H<sub>2</sub>O, hot EtOH. Insol. Et<sub>2</sub>O. *Ethochloride*: needles from EtOH-Et<sub>2</sub>O. M.p. 150° decomp. *Ethobromide*: prisms from H<sub>2</sub>O. M.p. 154°. *Ethiodide*: yellow plates from H<sub>2</sub>O or EtOH. M.p. 210° decomp.

*Et ester*: C<sub>13</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 215. Needles from dil. EtOH. M.p. 71°. *Methochloride*: yellow plates or needles from H<sub>2</sub>O or EtOH. M.p. 158° decomp. *Methiodide*: yellow plates or needles from H<sub>2</sub>O or EtOH. M.p. 208° decomp. Spar. sol. cold H<sub>2</sub>O, EtOH. *Ethochloride*: needles from EtOH-Et<sub>2</sub>O. M.p. 146° decomp. *Ethobromide*: prisms from H<sub>2</sub>O. M.p. 217°. *Ethiodide*: orange needles from EtOH. M.p. 236° decomp.

*Propyl ester*: C<sub>14</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 229. Prisms or needles from EtOH. M.p. 51°. *Methiodide*: yellow needles. M.p. 186° decomp.

*Benzyl ester*: C<sub>18</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 277. Prisms. M.p. 82°. *Methiodide*: yellow needles from H<sub>2</sub>O. M.p. 172°.

*Amide*: C<sub>11</sub>H<sub>10</sub>ON<sub>2</sub>. MW, 186. Cryst.

*Nitrile*: 3-cyanoquinaldine. C<sub>11</sub>H<sub>8</sub>N<sub>2</sub>. MW, 168. Prisms from H<sub>2</sub>O or needles from EtOH. M.p. 131° (125–7°).

Friedländer, Gohring, *Ber.*, 1883, **16**, 1836.

Hantzsch, *Ber.*, 1886, **19**, 37.

Claus, Steinitz, *Ann.*, 1894, **282**, 117 (Footnote).

Rohde, *Ber.*, 1889, **22**, 267.

v. Walther, *J. prakt. Chem.*, 1903, **67**, 509.

v. Meyer, *J. prakt. Chem.*, 1914, **90**, 27.

**Quinaldine-4-carboxylic Acid** (2-Methylquinoline-4-carboxylic acid, 2-methylcinchoninic acid, aniluvitonic acid).

Yellow cryst. +  $\text{H}_2\text{O}$  from hot  $\text{H}_2\text{O}$ . M.p.  $244^\circ$  ( $242^\circ$ ,  $246^\circ$  decomp.). Sol. hot EtOH, AcOH, dil. acids. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{CHCl}_3$ , pet. ether. Sublimes. Heat  $\longrightarrow$  quinaldine. Alk.  $\text{KMnO}_4 \longrightarrow$  6-methylpyridine-2:3:4-tricarboxylic acid. Acid  $\text{KMnO}_4 \longrightarrow$  acetylanthranilic acid.

*Me ester*: m.p.  $61-2^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Part. decomp. on dist.

*Et ester*: prisms from pet. ether. M.p.  $77^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . *Picrate*: yellow needles from EtOH. M.p.  $156^\circ$ .  $\text{B}_2\text{H}_2\text{PtCl}_6$ : yellow needles +  $2\text{H}_2\text{O}$ . M.p.  $203^\circ$  decomp.

*Amide*: needles from  $\text{H}_2\text{O}$ . M.p.  $239^\circ$ . Sol. hot EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Br + NaOH  $\longrightarrow$  4-aminoquinaldine. *Picrate*: m.p.  $239^\circ$  ( $231-2^\circ$  decomp.).

$\text{B}_2\text{H}_2\text{PtCl}_6$ : yellow needles +  $2\text{H}_2\text{O}$ . M.p.  $220^\circ$  decomp.

*Picrate*: greenish-yellow needles from EtOH. M.p.  $191^\circ$ .

v. Miller, *Ber.*, 1891, **24**, 1918.

Böttinger, *Ann.*, 1878, **191**, 321.

Simon, *Ann. chim. phys.*, 1896, **9**, 466.

Beyer, *J. prakt. Chem.*, 1886, **33**, 411.

Pfützing, *J. prakt. Chem.*, 1886, **33**, 100; 1888, **38**, 582; 1897, **56**, 284.

Bayer, D.R.P., 290,703, (*Chem. Zentr.*, 1916, I, 645).

Knövenagel, Bähr, *Ber.*, 1922, **55**, 1927.

**Quinaldine-5-carboxylic Acid** (2-Methylquinoline-5-carboxylic acid).

Needles from EtOH. M.p.  $285^\circ$  decomp. Sol. cold  $\text{NH}_3$  and warm dil. acids. Mod. sol. EtOH. Prac. insol.  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , ligroin.

*Nitrile*: 5-cyanoquinaldine. Needles +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $82^\circ$ , anhyd.  $104^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , hot  $\text{H}_2\text{O}$ . Volatile in steam.

Doebner, v. Miller, *Ber.*, 1884, **17**, 941.

Eckhardt, *Ber.*, 1889, **22**, 281.

v. Miller, *Ber.*, 1890, **23**, 2263.

Richard, *ibid.*, 3489.

Rist, *ibid.*, 3486.

**Quinaldine-6-carboxylic Acid** (2-Methylquinoline-6-carboxylic acid).

Needles from EtOH. M.p.  $261-2^\circ$  ( $259^\circ$ ,

$256^\circ$ ). Sol. hot EtOH. Spar. sol. hot  $\text{H}_2\text{O}$ . Sublimes.

Doebner, v. Miller, *Ber.*, 1884, **17**, 939.

v. Miller, *Ber.*, 1890, **23**, 2263.

I.G., D.R.P., 567,273, (*Chem. Zentr.*, 1933, I, 1687).

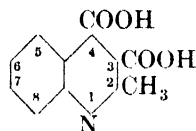
**Quinaldine-8-carboxylic Acid** (2-Methylquinoline-8-carboxylic acid).

Needles +  $\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $151^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH, acids, alkalis. Part. decomp. on heating  $\longrightarrow$  quinaldine.

Doebner, v. Miller, *Ber.*, 1884, **17**, 943.

v. Miller, *Ber.*, 1890, **23**, 2259.

**Quinaldine-3:4-dicarboxylic Acid** (2-Methylquinoline-3:4-dicarboxylic acid)



$\text{C}_{12}\text{H}_9\text{O}_4\text{N}$

MW, 231

Needles +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $238-9^\circ$  ( $236-7^\circ$ ) (slow heat.), about  $245^\circ$  (rapid heat.). Prac. insol.  $\text{H}_2\text{O}$ .

*Di-Et ester*:  $\text{C}_{16}\text{H}_{17}\text{O}_4\text{N}$ . MW, 287. M.p.  $88-9^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH.

*Anhydride*:  $\text{C}_{12}\text{H}_7\text{O}_3\text{N}$ . MW, 213. M.p.  $218^\circ$ .

*Imide*:  $\text{C}_{12}\text{H}_8\text{O}_2\text{N}_2$ . MW, 212. Yellow needles from AcOEt. M.p.  $257^\circ$ .

*3-Nitrile*: 3-cyanoquinaldine-4-carboxylic acid, 2-methyl-3-cyanocinchoninic acid. Plates from EtOH. M.p.  $238^\circ$  decomp. Sol. NaOH. Heat  $\longrightarrow$  3-cyanoquinaldine.

v. Walther, *J. prakt. Chem.*, 1903, **67**, 504.

Pfützing, *J. prakt. Chem.*, 1897, **56**, 316.

Engelhard, *J. prakt. Chem.*, 1898, **57**, 479.

Schering, D.R.P., 275,963, (*Chem. Zentr.*, 1914, II, 182).

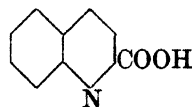
Lawson, Perkin, Robinson, *J. Chem. Soc.*, 1924, **125**, 634.

**Quinaldine-4:6-dicarboxylic Acid** (2-Methylquinoline-4:6-dicarboxylic acid).

Powder. Sinters at  $160^\circ$ .

v. Miller, *Ber.*, 1890, **23**, 2262.

**Quinaldinic Acid** (Quinaldic acid, quinoline-2-carboxylic acid).



$\text{C}_{10}\text{H}_7\text{O}_2\text{N}$

MW, 173

Needles + 2H<sub>2</sub>O from H<sub>2</sub>O. Loses H<sub>2</sub>O at 100°. M.p. anhyd. 157°. Sol. hot H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Mod. sol. cold H<sub>2</sub>O.  $k$  (acid) =  $1.2 \times 10^{-5}$  at 25°. Gives reddish-yellow col. with FeSO<sub>4</sub>. Heat  $\rightarrow$  quinoline. Alk. KMnO<sub>4</sub>  $\rightarrow$  pyridine-2 : 3 : 6-tricarboxylic acid. HNO<sub>3</sub> at 60-70°  $\rightarrow$  5- and 8-nitroquinaldinic acids. Used in estimation of Zn, Cu, Cd, UO<sub>2</sub>, and for separation of Cu from Cd, P, As, Pb, Ni, CO, Mn, etc.

*Me ester*: C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 187. Needles from ligroin. M.p. 86° (85°, 78°). *Methiodide*: orange cryst. M.p. 122-9° decomp. *Methochloride*: m.p. 158-65° decomp. *Methonitrate*: m.p. 138° decomp.

*Et ester*: C<sub>12</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 201. Needles. M.p. 36°.

*Nitrile*: 2-cyanoquinoline. C<sub>10</sub>H<sub>6</sub>N<sub>2</sub>. MW, 154. Needles. M.p. 94°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, Et<sub>2</sub>O.

*Amide*: C<sub>10</sub>H<sub>8</sub>ON<sub>2</sub>. MW, 172. Needles from dil. EtOH. or C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 133° (123°). Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOH. Spar. sol. Et<sub>2</sub>O, ligroin, hot H<sub>2</sub>O. Sol. dil. HCl.

*Chloride*: C<sub>10</sub>H<sub>6</sub>ONCl. MW, 191.5. Exists in two forms. (i) M.p. 175-6°: (ii) m.p. 97°.

*N-Oxide*: needles from H<sub>2</sub>O. M.p. 171° decomp. (167° decomp.). Sol. Me<sub>2</sub>CO, EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O, ligroin.

*Picrate*: yellow needles from H<sub>2</sub>O. Sol. hot H<sub>2</sub>O, EtOH.

Doebner, v. Miller, *Ber.*, 1883, **16**, 2472.

Besthorn, Ibele, *Ber.*, 1906, **39**, 2329.

v. Miller, Krämer, *Ber.*, 1891, **24**, 1915.

Besthorn, *Ber.*, 1909, **42**, 2698.

Meyer, *Monatsh.*, 1904, **25**, 1199.

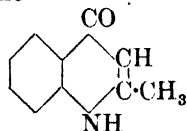
Mills, Hamer, *J. Chem. Soc.*, 1922, **121**, 2008.

Taylor, *J. Chem. Soc.*, 1929, 1110.

Hammick, *J. Chem. Soc.*, 1923, **123**, 2882.

Hammick, Dickinson, *J. Chem. Soc.*, 1929, 214.

Kaufmann, Dändliker, *Ber.*, 1913, **46**, 2928.

 $\gamma$ -QuinaldoneC<sub>10</sub>H<sub>8</sub>ON

MW, 159

The compound of the above formula described by Heller and Sourlis (*Ber.*, 1908, **41**, 2696) has been shown by Meisenheimer and Stotz (*Ber.*, 1925, **58**, 2334) to be quinaldine *N*-oxide.

*N-Me*: 1 : 2-dimethyl- $\gamma$ -quinolone. Needles

from C<sub>6</sub>H<sub>6</sub>. M.p. 176°. Sol. H<sub>2</sub>O, EtOH, hot C<sub>6</sub>H<sub>6</sub>. *B,HCl*: prisms + H<sub>2</sub>O. M.p. anhyd. 217°. *B,Hl*: needles + H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 201°. *B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow cryst. from H<sub>2</sub>O. M.p. 240° decomp. Sol. hot H<sub>2</sub>O. *Picrate*: decomp. at 233°.

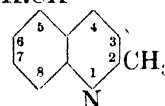
Conrad, Limpach, *Ber.*, 1887, **20**, 956.

Conrad, Eckhardt, *Ber.*, 1889, **22**, 76.

Knorr, *Ber.*, 1897, **30**, 925.

## 2-[5-Quinaldyl]-acrylic Acid

HOOC·CH:CH

C<sub>13</sub>H<sub>11</sub>O<sub>2</sub>N

MW, 213

Prisms from EtOH. M.p. 246° decomp. Mod. sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Prac. insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, ligroin. KMnO<sub>4</sub>  $\rightarrow$  quinaldine-5-aldehyde.

*Ag salt*: cryst. + 2 or 4H<sub>2</sub>O.

*B,HCl*: needles + H<sub>2</sub>O.

*B,HNO<sub>3</sub>*: needles + H<sub>2</sub>O.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow plates + 2H<sub>2</sub>O.

*Picrate*: needles + H<sub>2</sub>O from EtOH. M.p. 150-2°. Sol. hot H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO. Prac. insol. Et<sub>2</sub>O.

Eckhardt, *Ber.*, 1889, **22**, 272.

See also Decker, Remfry, *Ber.*, 1905, **38**, 2775.

## 2-[6-Quinaldyl]-acrylic Acid.

Needles from EtOH. M.p. 240-50°. Spar. sol. cold EtOH. Prac. insol. H<sub>2</sub>O. Sol. dil. alkalis. Spar. sol. dil. acids. KMnO<sub>4</sub>  $\rightarrow$  quinaldine-6-aldehyde.

*B,HCl*: prisms + H<sub>2</sub>O from dil. HCl. Sol. H<sub>2</sub>O.

*B,HNO<sub>3</sub>*: prisms + H<sub>2</sub>O. Spar. sol. dil. HNO<sub>3</sub>.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: reddish-yellow prisms + 2H<sub>2</sub>O.

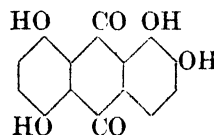
v. Miller, Kinkelin, *Ber.*, 1885, **18**, 3235.

## 2-[7-Quinaldyl]-acrylic Acid.

Cryst. +  $\frac{1}{2}$ EtOH from EtOH. M.p. 204°.

Eckhardt, *Ber.*, 1889, **22**, 273.

**Quinalizarin** (1 : 2 : 5 : 8-Tetrahydroxyanthraquinone)

C<sub>14</sub>H<sub>8</sub>O<sub>6</sub>

MW, 272

Red needles with green metallic lustre from  $\text{PhNO}_2$ . Does not melt below  $275^\circ$ . Very spar. sol. most solvents. Sublimes. Sol. alkalis with reddish-violet col. Sol. conc.  $\text{H}_2\text{SO}_4$  with bluish-violet col. Zn fusion  $\longrightarrow$  anthracene.

*Tetra-acetyl*: needles from  $\text{EtOH}-\text{CHCl}_3$ . M.p.  $201^\circ$ .

1:2-*Di-Me ether*:  $\text{C}_{16}\text{H}_{12}\text{O}_6$ . MW, 300. Brownish-red plates from  $\text{EtOH}$  or  $\text{C}_6\text{H}_6$ . M.p.  $225-30^\circ$ . Mod. sol. boiling  $\text{AcOH}$ . Spar. sol. hot  $\text{EtOH}$ , hot  $\text{C}_6\text{H}_6$ . Sol. alkalis with bluish-violet col. Sol. conc.  $\text{H}_2\text{SO}_4$  with blue col.

Liebermann, Kostanecki, *Ann.*, 1887, **240**, 301.

Schmidt, *J. prakt. Chem.*, 1891, **43**, 239.

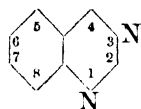
Gattermann, *ibid.*, 249.

Graebe, *Ber.*, 1890, **23**, 3739.

### Quinanisole.

See under 6-Hydroxyquinoline and 8-Hydroxyquinoline.

**Quinazoline** (5:6-Benzpyrimidine, phenmiazine)



$\text{C}_8\text{H}_6\text{N}_2$

MW, 130

Yellow plates from pet. ether. M.p.  $48-48.5^\circ$ . B.p.  $243^\circ/772.5\text{ mm.}$  ( $241.5^\circ/764\text{ mm.}$ ). Sol.  $\text{H}_2\text{O}$  with neutral reaction. Sol. usual solvents. Odour resembles naphthalene. Alk.  $\text{KMnO}_4 \longrightarrow$  pyrimidine-4:5-dicarboxylic acid + 4-hydroxyquinazoline.

*B,HAuCl*<sub>4</sub>: orange-red cryst. +  $\text{H}_2\text{O}$ . M.p.  $185^\circ$ .

*B*<sub>2</sub>, *H*<sub>2</sub>*PtCl*<sub>6</sub>: orange-yellow prisms. Does not melt below  $250^\circ$ .

*Picrate*: needles. M.p.  $188-90^\circ$ .

3-*Methoxyhydroxide*: prisms from  $\text{H}_2\text{O}$ . M.p.  $163-5^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{HCl}$ .

3-*Methochloride*: needles. Sinters at  $165-6^\circ$ , m.p.  $171-2^\circ$ . Sol.  $\text{H}_2\text{O}$ .

3-*Methobromide*: m.p.  $150-2^\circ$ . Sol.  $\text{H}_2\text{O}$ .

3-*Methiodide*: yellow prisms +  $\text{MeOH}$ . M.p.  $125-7^\circ$ . Sol.  $\text{H}_2\text{O}$ .

3-*Ethoxyhydroxide*: m.p.  $145-6^\circ$ . Sol. hot  $\text{H}_2\text{O}$ .

3-*Ethochloride*: m.p.  $150-1^\circ$ . Sol.  $\text{H}_2\text{O}$ .

Gabriel, *Ber.*, 1903, **36**, 808.

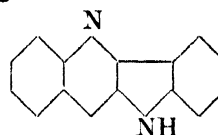
Bischer, Lang, *Ber.*, 1895, **28**, 292.

Riedel, D.R.P., 174,941, (*Chem. Zentr.*, 1906, II, 1372).

### Quinazolone.

See Hydroxyquinazoline.

### Quindoline



$\text{C}_{15}\text{H}_{10}\text{N}_2$

MW, 218

Needles from  $\text{EtOH}$ . M.p.  $247-8^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$  with blue fluor. Insol.  $\text{H}_2\text{O}$ . Sol. hot dil.  $\text{HCl}$ , conc.  $\text{H}_2\text{SO}_4$  with blue fluor. Sublimes with part. decomp.

*B,HCl*: yellow cryst. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*B,HI*: yellow cryst. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*B,HNO*<sub>3</sub>: yellow cryst. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*Picrate*: yellow cryst. Sol.  $\text{EtOH}$ .

*Methochloride*: yellow cryst. M.p.  $273^\circ$  decomp.

*Methiodide*: yellow needles from  $\text{H}_2\text{O}$ .

*Methosulphate*: orange-yellow needles. M.p.  $242-5^\circ$ .

*Ethiodide*: yellow needles from  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . M.p.  $222-3^\circ$ .

*Acetyl*: yellow needles from  $\text{EtOH}$ . M.p.  $177-8^\circ$ .

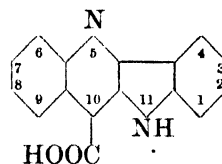
Fichter, Boehringer, *Ber.*, 1906, **39**, 3940.

Fichter, Rohner, *Ber.*, 1910, **43**, 3490.

Noelting, Steuer, *ibid.*, 3512.

Armit, Robinson, *J. Chem. Soc.*, 1922, **121**, 836.

### Quindoline-10-carboxylic Acid (Flavindin)



$\text{C}_{16}\text{H}_{10}\text{O}_2\text{N}_2$

MW, 262

Yellow ppt.  $\text{NaHg}$  or  $\text{Zn} \longrightarrow$  quindoline.

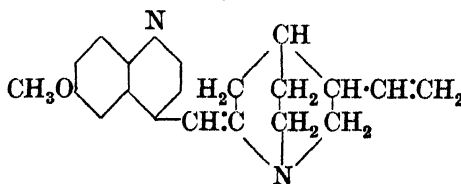
Giraud, *Compt. rend.*, 1879, **89**, 104; 1880, **90**, 1429.

Noelting, Steuer, *Ber.*, 1910, **43**, 3512.

Fichter, Rohner, *ibid.*, 3489.

Armit, Robinson, *J. Chem. Soc.*, 1922, **121**, 836.

### Quinene (Quinenine)



$\text{C}_{20}\text{H}_{22}\text{ON}_2$

MW, 306

Rhombic cryst. + 2H<sub>2</sub>O from Et<sub>2</sub>O or ligroin. Sinters at 75°, m.p. 81–2°. Loses H<sub>2</sub>O at 100°. 25% H<sub>3</sub>PO<sub>4</sub> at 170–80° → 6-methoxylepidine + meroquinene. HBr (D 1.49) at 190° → apoquinene. Br in CHCl<sub>3</sub> → quinene dibromide. Fluoresces in dil. H<sub>2</sub>SO<sub>4</sub>. Gives thalleioquin reaction.

*B,2HCl*: yellow needles + H<sub>2</sub>O from EtOH. M.p. 180–5°. [ $\alpha$ ]<sub>D</sub><sup>24</sup> (anhyd.) + 18.4° in H<sub>2</sub>O.

*B,2HCl,CuCl<sub>2</sub>*: yellowish-green cryst. Decomp. at 125–30°.

Comstock, Koenigs, *Ber.*, 1884, **17**, 1989; 1885, **18**, 1223.

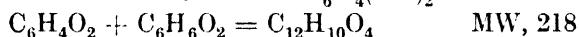
Heidelberger, Jacobs, *J. Am. Chem. Soc.*, 1920, **42**, 1501.

Cohen, *J. Chem. Soc.*, 1933, 996.

### Quinenine.

See Quinine.

**Quinhydrone** (*Molecular comp. of p-benzoquinone and hydroquinone*)



Reddish-brown needles with green lustre. M.p. 171°. Sol. EtOH, Et<sub>2</sub>O with yellow col. Spar. sol. cold H<sub>2</sub>O. Insol. pet. ether, ligroin. Sol. NH<sub>3</sub> with green col. D<sub>20</sub> 1.401. Sublimes. Boiling H<sub>2</sub>O → quinone + hydroquinone.

Bamberger, Czerkis, *J. prakt. Chem.*, 1903, **68**, 486.

Wöhler, *Ann.*, 1844, **51**, 153.

Torrey, Hardenbergh, *Am. Chem. J.*, 1905, **33**, 168.

Clark, *Am. Chem. J.*, 1892, **14**, 571.

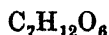
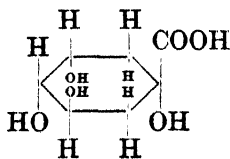
Michael, Cobb, *J. prakt. Chem.*, 1910, **82**, 304.

Evans, Dehn, *J. Am. Chem. Soc.*, 1930, **52**, 3204.

Müller, *Chem. Abstracts*, 1929, **23**, 5471.

Trénel, Bischoff, *Z. angew. Chem.*, 1929, **42**, 288.

**Quinic Acid** (1 : 3 : 4 : 5-Tetrahydroxycyclohexane-1-carboxylic acid, 1 : 3 : 4 : 5-tetrahydroxyhexahydrobenzoic acid)



MW, 192

*l.*

Occurs in cinchona bark, coffee beans, bilberries, sugar beet, etc. Prisms from H<sub>2</sub>O. M.p. 162°. Sol. 2.5 parts H<sub>2</sub>O at 9°. [ $\alpha$ ]<sub>D</sub><sup>15</sup> –43.84° in H<sub>2</sub>O, [ $\alpha$ ]<sub>D</sub><sup>20</sup> –44.03° in H<sub>2</sub>O, [ $\alpha$ ]<sub>D</sub><sup>25</sup> –42.1° in H<sub>2</sub>O. Triboluminescent. Heat at 200–50° →  $\gamma$ -lactone (quinide). KMnO<sub>4</sub> → *p*-benzoquinone. PbO<sub>2</sub> → hydroquinone. KOH fusion → protocatechuic acid. HI at 115–20° → benzoic acid.

*NH<sub>4</sub> salt*: cryst. from H<sub>2</sub>O. M.p. 179°.

*Na Salt*: prisms or plates + 2H<sub>2</sub>O. Sol. ½ part H<sub>2</sub>O at 15°.

*Tetra-acetyl*: cryst. M.p. 132.6°. Sol. EtOH, Et<sub>2</sub>O, alkalis. Mod. sol. H<sub>2</sub>O. Spar. sol. CS<sub>2</sub>. Insol. ligroin. [ $\alpha$ ]<sub>D</sub><sup>20</sup> –22.50° in EtOH.

*Tetrabenzoyl*: amorph. + 2H<sub>2</sub>O. M.p. 137–8°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Me ester*: C<sub>8</sub>H<sub>14</sub>O<sub>6</sub>. MW, 206. Needles from MeOH. M.p. 120° (126° to a milky liquid becoming clear at 142–3°). Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, pet. ether. *Tetra-Me ether*: m.p. 56–8°. [ $\alpha$ ]<sub>D</sub> –18.5° in C<sub>6</sub>H<sub>6</sub>.

*Ester*: C<sub>9</sub>H<sub>16</sub>O<sub>6</sub>. MW, 220. Viscous mass. Sol. H<sub>2</sub>O, EtOH. Less sol. Et<sub>2</sub>O. *Tetra-acetyl*: plates from H<sub>2</sub>O. M.p. 135–6°. Mod. sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Sublimes.

*Phenyl ester*: C<sub>13</sub>H<sub>16</sub>O<sub>6</sub>. MW, 268. *Tetra-acetyl*: cryst. from dil. EtOH. M.p. 167°. Sol hot EtOH, AcOH. Insol. cold H<sub>2</sub>O, Et<sub>2</sub>O.

*Amide*: C<sub>7</sub>H<sub>13</sub>O<sub>5</sub>N. MW, 191. Cryst. M.p. 132°. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. *Tetra-acetyl*: m.p. 186–7° decomp. [ $\alpha$ ]<sub>D</sub><sup>21</sup> –28.6° in tetrachloroethane. *Tetra-Me ether*: m.p. 115–16°.

*Nitrile*: C<sub>7</sub>H<sub>11</sub>O<sub>4</sub>N. MW, 173. *Tetra-acetyl*: m.p. 161–2°. [ $\alpha$ ]<sub>D</sub><sup>18</sup> –29.9° in CHCl<sub>3</sub>.

$\gamma$ -Lactone: see Quinide.

*d.*

Prisms. M.p. 164°. Sol. hot H<sub>2</sub>O. Spar. sol. EtOH. Prac. insol. Et<sub>2</sub>O. [ $\alpha$ ]<sub>D</sub><sup>20</sup> +44° in H<sub>2</sub>O.

*dl.*

Cryst.  $k = 2.2 \times 10^{-4}$  at 9°.

Fischer, Dangschat, *Ber.*, 1932, **65**, 1009  
Karrer, Widmer, Riso, *Helv. Chim. Acta* 1925, **8**, 195.

Herzig, Ortony, *Arch. Pharm.*, 1920, **258**, 91.

Gorter, *Ann.*, 1908, **358**, 329; 1908, **359**, 221.

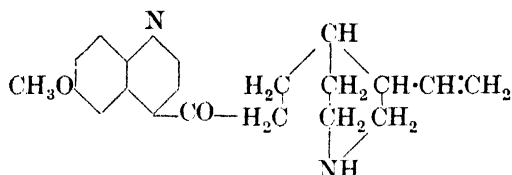
Erwig, Koenigs, *Ber.*, 1889, **22**, 1461.

Echtermeier, *Arch. Pharm.*, 1906, **244**, 42.

v. Lippemann, *Ber.*, 1901, **34**, 1159.

Hesse, *Ann.*, 1859, **110**, 333.

Zwenger, *Ann.*, 1860, **115**, 108.

**Quinicine** (*Quinotoxine*) $C_{20}H_{24}O_2N_2$ 

MW, 324

One of the cinchona alkaloids. Isomeric with quinine. Reddish-yellow amorph. mass. M.p. about  $60^\circ$ . Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Prac. insol. H<sub>2</sub>O. Sol. in EtOH absorbs CO<sub>2</sub>. Forms stable salts. Pptd. from sol. by excess KCNO.  $[\alpha]_D^{25} + 44.1^\circ$  in CHCl<sub>3</sub>. Shows no fluor. in dil. H<sub>2</sub>SO<sub>4</sub>. Gives thalleioquin reaction. *B, HCl*: leaflets. M.p.  $180-2^\circ$  ( $179-80^\circ$ ).  $[\alpha]_D^{25.7} + 16.26^\circ$  in H<sub>2</sub>O.

*B*<sub>2</sub>(COOH)<sub>2</sub>: prisms + 9H<sub>2</sub>O. M.p.  $149^\circ$ .

*Oxime*: m.p.  $112-16^\circ$ .

*p*-Bromophenylhydrazone: yellow cryst. M.p.  $141^\circ$ .

*N-Me*: yellow oil. *B, 2HCl*: cryst. + H<sub>2</sub>O. M.p.  $153-5^\circ$ .  $[\alpha]_D^{23} + 16.6^\circ$  in H<sub>2</sub>O.

*N-Et*: yellow oil. *B, HCl*: yellow cryst. from 95% EtOH. M.p.  $202-4^\circ$ .  $[\alpha]_D^{23} + 68.1^\circ$  in H<sub>2</sub>O.

*Dihydrate*: see Quinoticine.

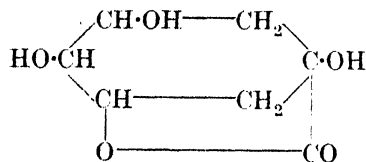
v. Miller, Rohde, Fussenegger, *Ber.*, 1900, **33**, 3228.

Howard, *J. Chem. Soc.*, 1871, **24**, 61, 1872, **25**, 101.

Hesse, *Ann.*, 1875, **178**, 244.

Fränkel, Diamant, *Ber.*, 1925, **58**, 554.

Heidelberger, Jacobs, *J. Am. Chem. Soc.*, 1919, **41**, 832; 1922, **44**, 1093.

**Quinide** ( $\gamma$ -Lactone of quinic acid) $C_7H_{10}O_5$ 

MW, 174

*dl*-

Cryst. from H<sub>2</sub>O or EtOH. M.p.  $200^\circ$  ( $198^\circ$ ). Sol. H<sub>2</sub>O. Spar. sol. dil. EtOH. Neutral reaction.

*l*-

M.p.  $187^\circ$ .  $[\alpha]_D^{17} - 17.13^\circ$  in H<sub>2</sub>O.

*Triacetyl*: prisms from EtOH. M.p.  $133-4^\circ$ .  $[\alpha]_D^{15} - 13.4^\circ$  in Me<sub>2</sub>CO.

*Tribenzoyl*: cryst. from EtOH. M.p.  $148^\circ$ .

Erwig, Koenigs, *Ber.*, 1889, **22**, 1458.

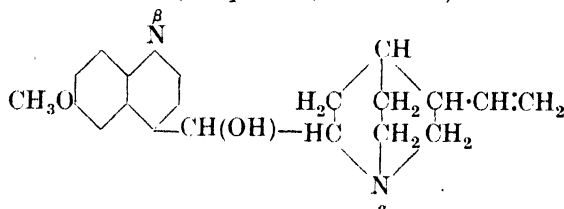
Hesse, *Ann.*, 1859, **110**, 335.

v. Lippmann, *Ber.*, 1901, **34**, 1159.

Echtermier, *Arch. Pharm.*, 1906, **244**, 53.

Gorter, *Ann.*, 1908, **359**, 223.

Fischer, *Ber.*, 1921, **54**, 781.

**Quinidine** (*Conquinine, conchinine*) $C_{20}H_{24}O_2N_2$ 

MW, 324

One of the cinchona alkaloids. Stereoisomeric with quinine. Cryst. +  $2\frac{1}{2}$ H<sub>2</sub>O from dil. EtOH. Loses  $\frac{1}{2}$ H<sub>2</sub>O on exposure to air. Cryst. + EtOH from EtOH. Loses EtOH at  $100^\circ$ . M.p. anhyd.  $174-5^\circ$  ( $171.5^\circ$ ,  $170-1^\circ$ ,  $168^\circ$ ).  $[\alpha]_D + 262^\circ$  ( $251^\circ$ ) in EtOH,  $[\alpha]_D^{15} + 236.8^\circ$  in 97% EtOH, +  $243.5^\circ$  in 99% EtOH.  $[\alpha]_D + 230^\circ$  in CHCl<sub>3</sub>. Sol. 6900 parts H<sub>2</sub>O at  $25^\circ$ , 750 parts at  $100^\circ$ . Sol. 26 parts EtOH, 22 parts Et<sub>2</sub>O at  $20^\circ$ . Anhyd. quinidine sol. 4950 parts H<sub>2</sub>O, 129 parts Et<sub>2</sub>O, 57 parts AcOEt, 177 parts CCl<sub>4</sub>, 41 parts C<sub>6</sub>H<sub>6</sub> at  $18-22^\circ$ . The salts of quinidine, with the exception of the hydriodide, are more sol. than those of quinine. Quinidine forms add. comps. with alcohols, acetone, benzene, etc. Cold acid KMnO<sub>4</sub>  $\rightarrow$  quitenidine + formic acid. Hot alk. KMnO<sub>4</sub>  $\rightarrow$  pyridine-2:3:4-tricarboxylic acid, oxalic acid, CO<sub>2</sub>, and NH<sub>3</sub>. CrO<sub>3</sub>  $\rightarrow$  quinone + quinic acid. PCl<sub>5</sub>  $\rightarrow$  quinidine chloride. Gives thalleioquin reaction. 60% H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  cupreidine + isocupreidine.

*B, H<sub>2</sub>SO<sub>4</sub>*: needles or prisms + 2H<sub>2</sub>O from hot H<sub>2</sub>O. Loses H<sub>2</sub>O at  $120^\circ$ . Sol. 108 parts H<sub>2</sub>O at  $10^\circ$ , 100 parts H<sub>2</sub>O at  $15^\circ$ , 7 parts H<sub>2</sub>O at  $100^\circ$ . Sol. 20 parts CHCl<sub>3</sub> at  $15^\circ$ . Sol. EtOH. Insol. Et<sub>2</sub>O.  $[\alpha]_D^{17} + 211.5^\circ$  in EtOH.  $[\alpha]_D^{15} + 218.2^\circ$  in 80% EtOH,  $[\alpha]_D^{15} + 179.5^\circ$  in H<sub>2</sub>O,  $[\alpha]_D + 184.17^\circ$  in CHCl<sub>3</sub>.

*B, H<sub>2</sub>SO<sub>4</sub>*: prisms + 4H<sub>2</sub>O. Sol. 8.7 parts H<sub>2</sub>O at  $10^\circ$  with blue fluor.

*B, HCl*: prisms + H<sub>2</sub>O. Sol. hot H<sub>2</sub>O, EtOH. Prac. insol. Et<sub>2</sub>O.  $[\alpha]_D^{15} + 200.9^\circ$  (+  $195.8^\circ$ ) in H<sub>2</sub>O. Also needles + 2H<sub>2</sub>O, m.p. anhyd.  $258-9^\circ$ . Sol. 75 parts H<sub>2</sub>O at  $15^\circ$ .  $[\alpha]_D + 212^\circ$  in EtOH,  $[\alpha]_D^{20} + 200^\circ$  in H<sub>2</sub>O.

*B, 2HCl*: prisms + H<sub>2</sub>O. Sol. EtOH. Spar. sol. CHCl<sub>3</sub>.  $[\alpha]_D^{15} + 250.3^\circ$  in H<sub>2</sub>O.

*B, HBr*: sol. 200 parts  $H_2O$  at  $14^\circ$ .

*B, HI*: prisms. Sol. 1270 parts  $H_2O$  at  $15^\circ$ .  
Prac. insol. EtOH.

*B, 2HI*: golden prisms +  $3H_2O$ . Sol. 90 parts  $H_2O$  at  $15^\circ$ .

*B, HNO<sub>3</sub>*: prisms. Sol. 85 parts  $H_2O$  at  $15^\circ$ .

*B, H<sub>3</sub>PO<sub>4</sub>*: prisms. Sol. 131 parts  $H_2O$  at  $10^\circ$ .  
Mod. sol. EtOH.

*B<sub>2</sub>, H<sub>2</sub>CrO<sub>4</sub>*: yellow plates +  $6H_2O$ .

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: orange needles +  $3H_2O$ .

*B<sub>2</sub>, 2HCl, CuCl<sub>2</sub>*: orange plates. M.p.  $208-9^\circ$  decomp.

*B<sub>2</sub>, (COOH)<sub>2</sub>*: cryst. +  $H_2O$  from  $H_2O$ . Sol. 151 parts  $H_2O$  at  $15^\circ$ .  $[\alpha]_D^{15} + 186.8^\circ$  in  $CHCl_3$ -EtOH.

*Benzoate*: cryst. +  $H_2O$ . Sol. EtOH.

*Salicylate*: sol. 1650 parts  $H_2O$  at  $25^\circ$ .

*Acetyl*: amorph. Sol. Et<sub>2</sub>O.  $[\alpha]_D^{15} + 128^\circ$  in EtOH. *B, H<sub>2</sub>AuCl<sub>4</sub>*: yellow anorph. +  $2H_2O$ . *B, H<sub>2</sub>PtCl<sub>6</sub>*: yellow cryst. +  $3H_2O$ . Spar. sol.  $H_2O$ .

*Salicyloyl*: powder. Sol. EtOH, Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ . *Salicylate*: needles from EtOH. M.p.  $168^\circ$ .

*$\alpha$ -Methiodide*: needles +  $H_2O$ . M.p.  $248^\circ$  decomp. Sol. hot  $H_2O$ , EtOH.

*Dimethiodide*: yellow plates +  $1\frac{1}{2}H_2O$ . M.p.  $156^\circ$  decomp. More sol.  $H_2O$  and less sol. EtOH than monomethiodide.

*$\alpha$ -Ethiodide*: m.p. anhyd.  $248^\circ$  decomp. Sol. hot  $H_2O$ , EtOH.

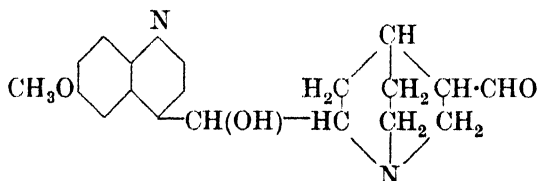
*Diethiodide*: yellow prisms +  $3H_2O$  from dil. EtOH. M.p. anhyd.  $205^\circ$  decomp. Sol. hot  $H_2O$ . Less sol. EtOH.

Hesse, *Ann.*, 1874, **174**, 337; *Ber.*, 1882, **15**, 3008.

Butler, Cretcher, *J. Am. Pharm. Assocn.*, 1933, **22**, 414.

Cohen, *J. Chem. Soc.*, 1933, 999.

### Quininal



$C_{19}H_{22}O_3N_2$

MW, 326

Cryst. M.p.  $160^\circ$ .  $[\alpha]_D^{15} - 30^\circ$  in  $CHCl_3$ .

*Phenylhydrazone*: m.p.  $145-7^\circ$ .

*p-Bromophenylhydrazone*: m.p.  $148-50^\circ$ .

*Acetyl*: m.p. about  $122^\circ$  decomp.  $[\alpha]_D^{25} - 63.5^\circ$  in  $CHCl_3$ .

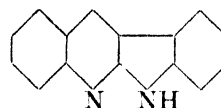
*Benzoyl*: m.p.  $126^\circ$ .  $[\alpha]_D^{25} + 79.4^\circ$  in  $CHCl_3$ .

*Chloropicrate*: m.p.  $130^\circ$ .

*Dichloropicrate*: m.p.  $126^\circ$  decomp.

Seekles, *Rec. trav. chim.*, 1923, **42**, 99.

### Quinindoline



$C_{15}H_{10}N_2$

MW, 218

Yellow needles or leaflets from  $PhNO_2$ . M.p.  $346^\circ$  ( $342-3^\circ$ ). Sol. boiling  $PhNO_2$ , aniline. Prac. insol. hot  $C_6H_6$ , AcOEt, EtOH,  $CHCl_3$ , Et<sub>2</sub>O. Sublimes. Sol.  $H_2SO_4$  with violet fluor.

*B, HCl*: yellow needles. M.p. about  $280^\circ$  decomp.

*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: yellow cryst.

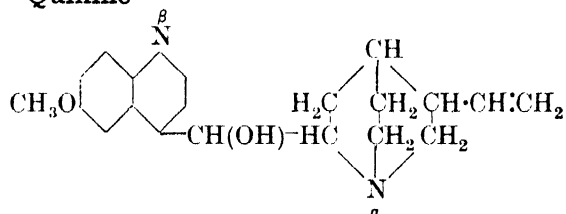
*Acetyl*: needles. M.p.  $185^\circ$ . Sol.  $C_6H_6$ . Spar. sol. MeOH.

Gabriel, Eschenbach, *Ber.*, 1897, **30**, 3020.

Lawson, Perkin, Robinson, *J. Chem. Soc.*, 1924, **125**, 634.

Friedländer, Sander, *Ber.*, 1924, **57**, 652.

### Quinine



$C_{20}H_{24}O_2N_2$

MW, 324

The most important of the cinchona alkaloids. Stereoisomeric with quinidine. Cryst. +  $3H_2O$  from Et<sub>2</sub>O below  $10^\circ$ , or needles +  $3H_2O$  from EtOH. Loses  $1H_2O$  above  $15^\circ$ ,  $2H_2O$  over  $H_2SO_4$ , and  $3H_2O$  at  $125^\circ$ . The commercial product is usually a microcryst. powder +  $2H_2O$ . *Trihydrate*: m.p.  $57^\circ$ : sol. 1670 parts  $H_2O$  at  $15^\circ$ , 0.8 part EtOH, 1.1 parts  $CHCl_3$ , 1.9 parts Et<sub>2</sub>O, 212 parts glycerol and 166 parts  $C_6H_6$  at  $25^\circ$ .  $[\alpha]_D^{15} - 145.2^\circ$  in EtOH. *Anhydrous*: m.p.  $177^\circ$  ( $176^\circ$ ,  $174.9^\circ$ ,  $172.8^\circ$ ): sol. 1960 parts  $H_2O$  at  $15^\circ$ , 760 parts  $H_2O$  at  $100^\circ$ , 1.1 parts EtOH, 22.6 parts Et<sub>2</sub>O, 1.9 parts  $CHCl_3$ , 200 parts  $C_6H_6$ , 189 parts  $CCl_4$  at  $18-22^\circ$ , 3450 parts 5% KOH and 1890 parts 10%  $NH_3$  at  $25^\circ$ . Sol. dil.  $H_2SO_4$  with blue fluor.  $[\alpha]_D^{15} - 158.7^\circ$  in Et<sub>2</sub>O,  $-169.3^\circ$  in 97% EtOH.  $[\alpha]_D^{17} - 167.5^\circ$  in EtOH,  $-117^\circ$  in  $CHCl_3$ . Bitter taste. Gives thalleioquin reaction. Acid  $KMnO_4$  in cold  $\rightarrow$  quitenine + formic acid. Alk.  $KMnO_4 \rightarrow$  pyridine-2 : 3 : 4-tricarboxylic acid.  $CrO_3 \rightarrow$  quinone + quinic acid.  $PCl_5$



—→ quinine chloride. Boiling AcOH —→ quinicine. Red. —→ hydroquinine. Br —→ quinine dibromide. HCl —→ apoquinine.  $\text{HNO}_3$  —→ cinchomeronic acid. KOH fusion —→ 6-methoxylepidine + 6-methoxyquinoline. Forms add. comp. with  $\text{C}_6\text{H}_6$  and other aromatic compounds. (Note: quinine is the Me ether of cupreine, but on demethylation apoquinine is formed, which is also obtained when cupreine is treated with hydrogen halides.)

$\text{B}_2\text{H}_2\text{SO}_4$ : silky needles +  $8\text{H}_2\text{O}$ . Bitter taste. Loses  $6\text{H}_2\text{O}$  on exposure to air giving dihydrate, m.p.  $205^\circ$ . Dehydrated completely at  $100^\circ$ . Sol. 740 parts  $\text{H}_2\text{O}$  at  $13^\circ$ , 30 parts  $\text{H}_2\text{O}$  at  $100^\circ$ , 65 parts EtOH at  $15^\circ$ , 24 parts glycerol at  $15^\circ$ , 1000 parts  $\text{CHCl}_3$  at  $15^\circ$ . Readily sol.  $\text{CHCl}_3$ -EtOH (2:1).  $[\alpha]_D^{15} = -157.4^\circ$  in EtOH.  $[\alpha]_D^{15}$  (anhyd.) —  $235^\circ$  in dil.  $\text{H}_2\text{SO}_4$ . Sol. dil.  $\text{H}_2\text{SO}_4$  with strong blue fluor. Pptd. by potassium chromate, oxalate or picrate, sodium salicylate, tannic acid, Rochelle salt, etc.

$\text{B}_2\text{H}_2\text{SO}_4$ : prisms +  $7\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$  or EtOH. Loses  $6\text{H}_2\text{O}$  over  $\text{H}_2\text{SO}_4$  and  $7\text{H}_2\text{O}$  at  $100^\circ$ . Softens at  $60^\circ$ , m.p. anhyd.  $160^\circ$ . Bitter taste. Shows strong fluor. in aq. sol. Sol. 11 parts  $\text{H}_2\text{O}$ , 45 parts EtOH at  $13^\circ$ , 18 parts glycerol, 920 parts  $\text{CHCl}_3$ , 1770 parts  $\text{Et}_2\text{O}$  at  $25^\circ$ .  $[\alpha]_D^{15} = -164.5^\circ$  in  $\text{H}_2\text{O}$ .

$\text{B}_2\text{H}_2\text{SO}_4$ : prisms +  $7\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ . Also prisms +  $5\text{H}_2\text{O}$  from EtOH.  $[\alpha]_D^{15} = -168.4^\circ$  in  $\text{H}_2\text{O}$ .

$\text{B}_2\text{HCl}$ : needles +  $2\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  at  $100^\circ$ . M.p. anhyd.  $158-60^\circ$ . Sol. 40 parts  $\text{H}_2\text{O}$ , 9 parts  $\text{CHCl}_3$ , 3 parts EtOH at  $15^\circ$ . Sol. 1 part boiling  $\text{H}_2\text{O}$ .  $[\alpha]_D^{17} = -133.7^\circ$  in  $\text{H}_2\text{O}$ , —  $57.1^\circ$  in  $\text{CHCl}_3$ .  $[\alpha]_D^{15} = -145.5^\circ$  in 97% EtOH.

$\text{B}_2\text{HCl}$ : needles. Turns brown at  $165-75^\circ$ , m.p.  $180-5^\circ$ . Sol. 0.75 part  $\text{H}_2\text{O}$ , 5 parts EtOH, 7 parts  $\text{CHCl}_3$  at  $15^\circ$ . Insol.  $\text{Et}_2\text{O}$ .  $[\alpha]_D^{17-19} = -233^\circ$  in  $\text{H}_2\text{O}$ .

$\text{B}_2\text{HBr}$ : needles +  $\text{H}_2\text{O}$ . Softens at  $152^\circ$ , melting finally at  $200^\circ$ . Sol. 55 parts  $\text{H}_2\text{O}$ , 7 parts EtOH, 10 parts  $\text{CHCl}_3$  at  $15^\circ$ . Sol. 1 part boiling  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Hygroscopic.

$\text{B}_2\text{HBr}$ : yellow prisms +  $3\text{H}_2\text{O}$ . M.p.  $81-2^\circ$ . Sol. 7 parts  $\text{H}_2\text{O}$  at  $15^\circ$ . Sol. EtOH. Insol.  $\text{Et}_2\text{O}$ .

$\text{B}_2\text{HI}$ : yellow needles. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

$\text{B}_2\text{HI}$ : yellow prisms +  $5\text{H}_2\text{O}$ . M.p. about  $100^\circ$ . Sol. 20 parts  $\text{H}_2\text{O}$  at  $15^\circ$ .

$\text{B}_2\text{HNO}_3$ : cryst. +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . Sol. 70 parts  $\text{H}_2\text{O}$  at  $25^\circ$ .

$\text{B}_2\text{H}_2\text{CO}_3$ : needles +  $\text{H}_2\text{O}$ . Sol. EtOH.

$\text{B}_2\text{H}_3\text{PO}_4$ : needles +  $8\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . Sol. 784 parts  $\text{H}_2\text{O}$  at  $10^\circ$ .

$\text{B}_2\text{H}_3\text{PO}_3$ : sol. 60 parts  $\text{H}_2\text{O}$  at  $15^\circ$ .

$\text{B}_2\text{H}_2\text{PtCl}_6$ : orange, amorph. +  $3\text{H}_2\text{O}$ . Prac. insol. cold  $\text{H}_2\text{O}$ .

$\text{B}_2\text{H}_2\text{PtCl}_6$ : yellow cryst. +  $\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  at  $100^\circ$ . Decomp. above  $100^\circ$ .

$\text{B}_2\text{H}_2\text{CrO}_4$ : yellow cryst. +  $2\text{H}_2\text{O}$ . Sol. 2400 parts  $\text{H}_2\text{O}$  at  $15^\circ$ , 160 parts  $\text{H}_2\text{O}$  at  $100^\circ$ . Sol. EtOH. Insol.  $\text{Et}_2\text{O}$ .

$\text{B}_2\text{HCl}, \text{CuCl}_2$ : brick-red needles. M.p.  $210^\circ$  decomp.

Formate:  $\text{B}_2\text{H}\cdot\text{COOH}$ . Quinaform. Needles. M.p.  $109^\circ$  decomp. Sol. 19 parts  $\text{H}_2\text{O}$  at  $16^\circ$ , 8 parts  $\text{H}_2\text{O}$  at  $32^\circ$ .  $[\alpha]_D^{20} = -144.2^\circ$  in  $\text{H}_2\text{O}$ .  $\text{B}_2\text{H}\cdot\text{COOH}$ : needles. Decomp. at  $50^\circ$ .

Acetate:  $\text{B}_2\text{CH}_3\text{COOH}$ . Needles. M.p.  $140^\circ$ . Sol. 30 parts  $\text{H}_2\text{O}$  at  $15^\circ$ .

Chloroacetate: cryst. +  $2\frac{1}{2}\text{H}_2\text{O}$ . Sol. 64 parts  $\text{H}_2\text{O}$  at  $21^\circ$ . Sol. hot EtOH.

Dichloroacetate: needles +  $2\text{H}_2\text{O}$ . Sol. 41.4 parts  $\text{H}_2\text{O}$  at  $22^\circ$ .

Trichloroacetate: needles. M.p.  $139-40^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH.

Oxalate:  $\text{B}_2(\text{COOH})_2$ . Prisms +  $6\text{H}_2\text{O}$ . Sol. 1030 parts  $\text{H}_2\text{O}$  at  $10^\circ$ .

d-Tartrate:  $\text{B}_2\text{C}_4\text{H}_6\text{O}_6$ . Cryst. powder +  $2\text{H}_2\text{O}$ . Loses  $1\text{H}_2\text{O}$  at  $120^\circ$  and second  $\text{H}_2\text{O}$  at  $140^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ .  $[\alpha]_D^{17} = -216.6^\circ$  in dil. HCl.  $\text{B}_2\text{C}_4\text{H}_6\text{O}_6$ : cryst. +  $\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  at  $160^\circ$ . Less sol.  $\text{H}_2\text{O}$  than l-tartrate.

l-Tartrate:  $\text{B}_2\text{C}_4\text{H}_6\text{O}_6$ . Cryst. +  $\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  at  $100^\circ$ . More sol.  $\text{H}_2\text{O}$  than d-tartrate.

Citrate:  $\text{B}_2\text{C}_6\text{H}_8\text{O}_7$ : needles from  $\text{H}_2\text{O}$ . M.p.  $204^\circ$  part decomp.  $\text{B}_2\text{C}_6\text{H}_8\text{O}_7$ . Prisms or needles +  $7\text{H}_2\text{O}$ . Sol. 930 parts  $\text{H}_2\text{O}$  at  $12^\circ$ .  $\text{B}_2\text{C}_6\text{H}_8\text{O}_7$ : cryst.

l-Lactate:  $\text{B}_2\text{C}_3\text{H}_6\text{O}_3$ . Needles +  $\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .

dl-Lactate:  $\text{B}_2\text{C}_3\text{H}_6\text{O}_3$ . Cryst. +  $\text{H}_2\text{O}$ .

Benzoate:  $\text{B}_2\text{C}_7\text{H}_6\text{O}_2$ . Prisms. Sol. 373 parts  $\text{H}_2\text{O}$  at  $100^\circ$ .

Salicylate:  $\text{B}_2\text{C}_7\text{H}_6\text{O}_3$ . Prisms from EtOH. Sol. 225 parts  $\text{H}_2\text{O}$  at  $16^\circ$ . Also cryst. +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. about  $195^\circ$ .

Acetylsalicylate:  $\text{B}_2\text{C}_9\text{H}_8\text{O}_4$ . Xaxaquin. M.p.  $157^\circ$ . Insol.  $\text{Et}_2\text{O}$ .

Acetyl: cryst. from pet. ether. M.p.  $116-17^\circ$  ( $108^\circ$ ). Sol. EtOH,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ . Almost tasteless.  $[\alpha]_D^{15} = -54.3^\circ$  in 97% EtOH,  $[\alpha]_D^{22.9} = -120.8^\circ$  in dil. HCl. Sol. dil. acids.

Propionyl: prisms from  $\text{Et}_2\text{O}$ . M.p.  $129^\circ$ . Sol.  $\text{CHCl}_3$ . Mod. sol. EtOH,  $\text{Et}_2\text{O}$ . Prac. insol.  $\text{H}_2\text{O}$ .

Isovaleryl: amorph. Hygroscopic. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

Benzoyl: prisms from  $\text{Et}_2\text{O}$ . M.p.  $139^\circ$  ( $138^\circ$ ). Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether,

$\text{CS}_2$ . Insol.  $\text{H}_2\text{O}$ . Sol. min. acids.  $[\alpha]_D^{17} + 121.6^\circ$  in EtOH,  $[\alpha]_D^{16.2} + 119.9^\circ$  in EtOH.

*Salicyloyl*: saloquinine. Cryst. from dil. EtOH or Et<sub>2</sub>O. M.p.  $140^\circ$ . Sol. EtOH, Et<sub>2</sub>O,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . *Salicylate*:  $\text{B}_2\text{C}_7\text{H}_6\text{O}_3$ . Rheumatism. Needles. M.p.  $179^\circ$ . Tasteless. Spar. sol.  $\text{H}_2\text{O}$ .

*Anisoyl*: needles from Et<sub>2</sub>O. M.p.  $87-8^\circ$ . Sol. EtOH,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

*Cinnamoyl*: needles from Et<sub>2</sub>O. M.p.  $111^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

$\alpha$ -*Methochloride*: needles +  $\text{H}_2\text{O}$ . M.p.  $181-2^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH.

$\alpha$ -*Methobromide*: needles +  $\text{H}_2\text{O}$ . M.p.  $124-6^\circ$ . Sol. EtOH. Mod. sol. cold  $\text{H}_2\text{O}$ .

$\alpha$ -*Methiodide*: needles + 1 or 2  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $233-6^\circ$  decomp. Sol. EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ . Prac. insol. Et<sub>2</sub>O,  $\text{CHCl}_3$ .

*Dimethiodide*: yellow plates + 3  $\text{H}_2\text{O}$ . M.p.  $167-8^\circ$  decomp. ( $158-62^\circ$  decomp.).  $[\alpha]_D^{18} - 151^\circ$  in 2N  $\text{H}_2\text{SO}_4$ .

$\alpha$ -*Ethochloride*: needles + 3  $\text{H}_2\text{O}$ .  $[\alpha]_D - 122^\circ$  in  $\text{H}_2\text{O}$ .

$\alpha$ -*Ethobromide*: cryst. + 2  $\text{H}_2\text{O}$ .  $[\alpha]_D - 117^\circ$  in  $\text{H}_2\text{O}$ .

$\alpha$ -*Ethiodide*: cryst. +  $\text{H}_2\text{O}$ . M.p. anhyd.  $210-11^\circ$ .  $[\alpha]_D - 105^\circ$  in EtOH.

$\beta$ -*Ethiodide*: needles + 3  $\text{H}_2\text{O}$ . M.p.  $93^\circ$ .

*Diethiodide*: yellow cryst. + 3  $\text{H}_2\text{O}$  from dil. EtOH. M.p.  $140^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH. Insol. Et<sub>2</sub>O.

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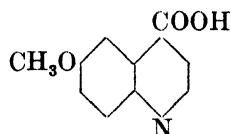
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**Quinic Acid** (6-Methoxycinchoninic acid, 6-hydroxyquinoline-4-carboxylic acid methyl ether)



$\text{C}_{11}\text{H}_9\text{O}_3\text{N}$

MW, 203

Yellow prisms from dil. HCl. M.p.  $280^\circ$  decomp. Sol. acids with yellow col. Sol. alkalis. Spar. sol. EtOH with blue fluor. destroyed by  $\text{H}_2\text{O}$  or acids. Spar. sol. cold  $\text{H}_2\text{O}$ , Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ .  $k = 9 \times 10^{-6}$  at  $25^\circ$ . Alk.

Dict. of Org. Comp.—III.

$\text{KMnO}_4 \rightarrow$  pyridine-2 : 3 : 4-tricarboxylic acid. Conc. HCl  $\rightarrow$  6-hydroxycinchoninic acid.

*Me ester*:  $\text{C}_{12}\text{H}_{11}\text{O}_3\text{N}$ . MW, 217. Prisms from EtOH or Et<sub>2</sub>O. M.p.  $85^\circ$ .

*Et ester*:  $\text{C}_{13}\text{H}_{13}\text{O}_3\text{N}$ . MW, 231. M.p.  $69^\circ$ . Insol.  $\text{H}_2\text{O}$ . *B, HCl*: yellow needles from EtOH. M.p.  $160^\circ$  decomp.  $\text{B}_2\text{H}_2\text{PtCl}_6$ : orange cryst. + 2  $\text{H}_2\text{O}$ . M.p.  $228^\circ$  decomp.

*Amide*:  $\text{C}_{11}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 202. Needles from AcOEt. M.p.  $197^\circ$ . Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ , Et<sub>2</sub>O. Insol. ligroin,  $\text{C}_6\text{H}_6$ .

*Chloride*:  $\text{C}_{11}\text{H}_9\text{O}_2\text{NCl}$ . MW, 221.5. *B, HCl*: yellow powder. Decomp. at  $186^\circ$ .

*Nitrile*: 6-methoxy-4-cyanoquinoline.  $\text{C}_{11}\text{H}_8\text{ON}_2$ . MW, 184. Yellow needles from  $\text{C}_6\text{H}_6$ . M.p.  $157^\circ$ . Readily sol. EtOH,  $\text{CHCl}_3$ , toluene. Sol. Et<sub>2</sub>O, ligroin. Insol.  $\text{H}_2\text{O}$ .

*Hydrazide*: m.p.  $151^\circ$ .

*B, HCl*: yellow plates + 2  $\text{H}_2\text{O}$ . M.p.  $225^\circ$ .

*Methochloride*: yellow needles. M.p.  $215^\circ$ . Sol.  $\text{H}_2\text{O}$ .

*Methiodide*: yellow plates or needles from EtOH or  $\text{H}_2\text{O}$ . M.p.  $205^\circ$  decomp. Sol. hot.  $\text{H}_2\text{O}$ , EtOH.

*Ethobromide*: yellow needles from EtOH. M.p.  $210^\circ$ .

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Hirsch, *Monatsh.*, 1896, 17, 327.

Koenigs, Schönewald, *Ber.*, 1902, 35, 2986.

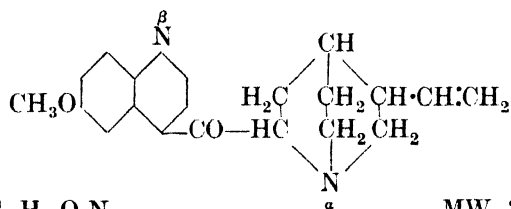
Kaufmann, Peyer, *Ber.*, 1912, 45, 1805.

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John, Andraschko, *J. prakt. Chem.*, 1930, 128, 180.

Rabe, Huntenburg, Schultze, Volger, *Ber.*, 1931, 64, 2492.

**Quininone** (6-Methoxycinchoninone)



$\text{C}_{20}\text{H}_{22}\text{O}_2\text{N}_2$

MW, 322

Needles or leaflets. M.p.  $108^\circ$  ( $101-8^\circ$  slow heat.).  $[\alpha]_D^{23} + 73.8^\circ$  in EtOH. Sol. EtOH, Et<sub>2</sub>O,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin.

*B, HCl*: hygroscopic cryst. M.p.  $210-12^\circ$ .  $[\alpha]_D^{14} + 58.7^\circ$  in EtOH.

*Oxime*: m.p.  $113^\circ$  (not sharp).

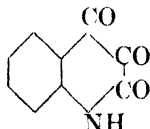
*Picrate*: cryst. from EtOH. M.p.  $232-3^\circ$ .

*Picolonate*: yellow needles from EtOH. M.p.  $197-8^\circ$ .

$\alpha$ -Methiodide: cryst. from MeOH. M.p. 213–14°.

Rabe, Kuliga, *Ann.*, 1909, **364**, 346, 349.

**Quinisatin** (2 : 3 : 4-Triketotetrahydroquinoline)



$C_9H_5O_3N$

MW, 175

Yellow prisms +  $H_2O$  from  $H_2O$ . Loses  $H_2O$  at 120–5° turning red. M.p. 255–60°. Mod. sol. cold, sol. hot  $H_2O$ . Anhyd. sol. EtOH. Sol. NaOH aq. with yellow col.  $NH_2OH \rightarrow$  3-oxime.

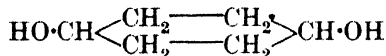
N-Me: reddish-yellow cryst. M.p. 120–2°. Sol. alkalis with yellow col. Oxime: red needles from AcOH. Decomp. at 188°. Sol. alkalis with green col.

3-Oxime: orange-yellow prisms from EtOH. M.p. 208° decomp. Sol. AcOH. Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Sol. alkalis with reddish-brown col. Sol.  $H_2SO_4$  with red col.  $Sn + HCl \rightarrow$  3-amino-2 : 4-dihydroxyquinoline.

Baeyer, Homolka, *Ber.*, 1883, **16**, 2219; 1884, **17**, 985.

Friedländer, Müller, *Ber.*, 1887, **20**, 2015. See also Kalb, *Ber.*, 1911, **44**, 1460.

**Quinitol** (Hexahydrohydroquinone, cyclohexandiol-1 : 4, 1 : 4-dihydroxycyclohexane)



$C_6H_{12}O_2$

MW, 116

*Cis* :

Prisms from  $Me_2CO$ . M.p. 102°. Sol.  $H_2O$ , EtOH,  $Me_2CO$ . Spar. sol.  $Et_2O$ ,  $CHCl_3$ . Sublimes in vacuo. Stable to  $KMnO_4$ .

Diacetyl: cryst. from dil. EtOH. M.p. 34–6°. B.p. 245–50°/710 mm., 145–7°/25 mm.

Dipropionyl: m.p. 39.5–40°.

Dibenzoyl: m.p. 116–17°.

Dicinnamoyl: m.p. 122°.

Me ether:  $C_7H_{14}O_2$ . MW, 130. B.p. 102–3°/15 mm.  $n_D^{20}$  1.4671.  $D^{20}$  1.023.

Di-Me ether:  $C_8H_{16}O_2$ . MW, 144. B.p. 67.5–68°/14 mm.  $n_D^{18}$  1.4440.  $D^{18}$  0.9526.

*Trans* :

Plates from  $Me_2CO$ . M.p. 139°. Sol.  $H_2O$ , EtOH. Spar. sol. cold  $Me_2CO$ . Very spar. sol.  $Et_2O$ .

Diacetyl: cryst. from EtOH. M.p. 102–3°. B.p. 245–50°/710 mm., 145–7°/25 mm.

Dipropionyl: m.p. 75.5–76°.

Dibenzoyl: m.p. 151°.

Dicinnamoyl: m.p. 189°.

Me ether: b.p. 102.5–103°/15 mm.  $n_D^{20}$  1.4649.  $D^{20}$  1.021.

Di-Me ether: b.p. 68–9°/15 mm.  $n_D^{18}$  1.4430.

Sabatier, Mailhe, *Ann. chim. phys.*, 1909, **16**, 90.

Leroux, *Ann. chim. phys.*, 1910, **21**, 542.

Baeyer, *Ann.*, 1894, **278**, 92.

Willstätter, Lessing, *Ber.*, 1901, **34**, 507.

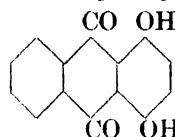
Uspenski, Turin, *Chem. Zentr.*, 1923, III, 754.

Palfray, Sabetay, *Bull. soc. chim.*, 1928, **43**, 898.

Palfray, Rothstein, *Bull. soc. chim.*, 1929, **45**, 855.

Rothstein, *Ann. chim.*, 1930, **14**, 486.

**Quinizarin** (1 : 4-Dihydroxyanthraquinone)



$C_{14}H_8O_4$

MW, 240

Red. cryst. from AcOH, m.p. 200–2°; red cryst. from toluene, m.p. 194°. Sol. 12–13 parts boiling AcOH. Sol. alkalis with violet-blue col. Sol. conc.  $H_2SO_4$  with violet-red col. and greenish-yellow fluor.  $PbO_2$  in AcOH  $\rightarrow$  anthra-1 : 4 : 9 : 10-diquinone.  $HNO_3 \rightarrow$  2-nitro deriv.  $K_3Fe(CN)_6 \rightarrow$  phthalic acid. Hydrosulphite  $\rightarrow$  leucoquinizarin. Intermediate for dyestuffs.

Me ether:  $C_{15}H_{10}O_4$ . MW, 254. Yellow needles from MeOH. M.p. 189° (167–8°). Sol. alkalis.

Di-Me ether:  $C_{16}H_{12}O_4$ . MW, 268. Cryst. from  $C_6H_6$ . M.p. 170–1° (143°).

Et ether:  $C_{16}H_{12}O_4$ . MW, 268. Red needles from EtOH. M.p. 150–1°. Mod. sol. alkalis with reddish-violet col.

Di-Et ether:  $C_{18}H_{16}O_4$ . MW, 296. Yellow needles. M.p. 176–7°.

Diphenyl ether: 1 : 4-diphenoxyanthraquinone.  $C_{26}H_{16}O_4$ . MW, 392. Yellow needles from EtOH. M.p. 165°. Sol. boiling EtOH,  $C_6H_6$ , AcOH. Spar. sol. boiling MeOH. Insol. ligroin. Sol. conc.  $H_2SO_4$  with bluish-violet col. Acetyl: orange-yellow needles from  $C_6H_6$ . M.p. 186°.

Diacetyl: orange-yellow cryst. from AcOH. M.p. 207–8° (200°). Exists in polymorphic forms.

1-Acetyl-4-benzoyl : m.p. 195-6°.

Baeyer, Caro, *Ber.*, 1875, **8**, 152.

Liebermann, *Ann.*, 1882, **212**, 10.

Bayer, D.R.P., 229,316, (*Chem. Zentr.*, 1911, **I**, 181); D.R.P., 255,031, (*Chem. Zentr.*, 1913, **I**, 354).

M.L.B., D.R.P., 242,379, (*Chem. Zentr.*, 1912, **I**, 301).

Eckert, Steiner, *Monatsh.*, 1914, **35**, 1145.

Green, *J. Chem. Soc.*, 1926, 1428.

Bigelow, Reynolds, *Organic Syntheses*, Collective Vol. I, 464.

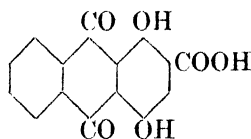
National Aniline and Chemical Co., U.S.P., 1,886,237, (*Chem. Abstracts*, 1933, **27**, 1366).

British Celanese, E.P., 346,355, (*Chem. Abstracts*, 1932, **26**, 1948).

United Alkali, E.P., 245,584, (*Chem. Abstracts*, 1927, **21**, 249).

du Pont, U.S.P., 2,003,859, (*Chem. Abstracts*, 1935, **29**, 4776).

**Quinizarin-2-carboxylic Acid** (1:4-Dihydroxyanthraquinone-2-carboxylic acid)



$C_{15}H_8O_6$

MW, 284

Red or yellowish-brown needles from  $PhNO_2$ . M.p. 249-50° (244-6°). Sol.  $Me_2CO$ , warm AcOH, toluene. Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Sol. alkalis. Heat  $\rightarrow$  quinizarin.

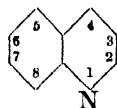
Ullmann, Schmidt, *Ber.*, 1919, **52**, 2111.

Bayer, D.R.P., 273,341, (*Chem. Zentr.*, 1914, **I**, 1719).

### Quinol.

See Hydroquinone.

**Quinoline** (2:3-Benzpyridine)



$C_9H_7N$

MW, 129

Occurs in coal-tar and in "stupp" fat. F.p. -20° (-15°). B.p. 238.05°/760 mm., 236.9°/749.2 mm., 114°/17 mm. Sol. hot  $H_2O$ , dil. acids, EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CS_2$ . Mod. sol. cold  $H_2O$ . Hygroscopic. Volatile in steam.  $D_4^{20}$  1.1081,  $D_5^{20}$  1.0947,  $D_6^{20}$  1.0929,  $D_7^{20}$  1.0900 (1.08979),  $D_8^{20}$  1.0699.  $n_D^{25}$  1.6305,  $n_D^{20}$  1.6268,  $n_D^{24}$  1.6245,  $n_D^{20}$  1.6218. Heat of comb.  $C_v$  1122.3 Cal.  $KMnO_4 \rightarrow$  quinolinic acid,

oxalic acid,  $NH_3$ , and  $CO_2$ .  $HNO_3 + H_2SO_4 \rightarrow$  5- + 8-nitroquinolines  $\rightarrow$  5:7- + 6:8-dinitroquinolines.  $H_2SO_4$  at 220°  $\rightarrow$  quinoline-8-sulphonic acid + a small quantity of quinoline-5-sulphonic acid.  $H_2SO_4$  at 300°  $\rightarrow$  quinoline-6-sulphonic acid.  $Sn + HCl$  or  $Na + EtOH \rightarrow$  1:2:3:4-tetrahydroquinoline.  $HI(+P) \rightarrow$  hexa- and decahydroquinolines.  $H(+Ni) \rightarrow$  1:2:3:4-tetrahydroquinoline  $\rightarrow$  decahydroquinoline.

$B, HCl$ : deliquescent prisms +  $\frac{1}{2}H_2O$ . M.p. 94°, anhyd. 134-5°. Sol. EtOH,  $CHCl_3$ . Spar. sol.  $Et_2O-C_6H_6$ .

$B, 3HCl$ : m.p. 82°.

$B, 2HCl$ : m.p. 46.7° (48.5-57°).

$B, HBr$ : cryst. +  $2H_2O$ . M.p. 41°.

$B, HI$ : yellow needles from EtOH. M.p. 135°.

$B, H_2SO_4$ : cryst. from EtOH or AcOH. M.p. 163.5-164.5°.

$B, HNO_3$ : needles from EtOH.

$B, HClO_3$ : yellow cryst. M.p. 66-7°. Hygroscopic. Sol. EtOH.

$B, HSCN$ : plates. M.p. 140°. Spar. sol.  $H_2O$ , EtOH.

$B, H_2Cr_2O_7$ : yellow needles from  $H_2O$ . M.p. 167° (165-7°). Sol. 282 parts  $H_2O$  at 10-5°.

$B, HCl, CuCl_2$ : brown needles. Decomp. at 185°.

$B, 2HCl, AuCl_3$ : cryst. ppt. M.p. 180°. Decomp. at about 260°.

$B, AuCl_3$ : yellow cryst. from EtOH.

$B, HAuCl_4$ : yellow needles. M.p. 235-8°. Decomp. at about 260°. Spar. sol. cold  $H_2O$ .

$B, H_2PtCl_6$ : orange-yellow needles +  $2H_2O$  from dil. HCl. M.p. 227.5° (225°, 226°, 218°).

$B, H_2PtBr_6$ : red prisms or needles. M.p. 254-5°.

Dichloroacetate: cryst. M.p. 63-4°. Sol.  $H_2O$ .

Trichloroacetate: cryst. from EtOH. M.p. 100°. Sol.  $H_2O$ .

$B, (COOH)_2$ : needles from EtOH. M.p. about 105°.

d-Tartrate: cryst. +  $5H_2O$ . M.p. 131°.  $[\alpha]_D^{20} + 13.2^\circ$  in  $H_2O$  ( $[\alpha]_D^{10} + 13.5^\circ$  in  $H_2O$ ).

Acid d-Tartrate:  $[\alpha]_D^{25} + 14.9^\circ$  in  $H_2O$ .

Acid phthalate: cryst. M.p. 98-9°. Sol. EtOH.

Picrate: yellow needles from  $C_6H_6$ . M.p. 203°.

Styphnate: m.p. 207-8°.

Methochloride: cryst. +  $H_2O$  from EtOH. M.p. 126°.

Methobromide: needles. M.p. 70°. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol.  $Me_2CO$ . Prac. insol.  $C_6H_6$ , pet. ether.

**Methiodide**: orange-red cryst. from EtOH, m.p. 133° (144·5°); yellow cryst. + H<sub>2</sub>O from EtOH, m.p. 72°; yellow cryst. + C<sub>6</sub>H<sub>6</sub> from C<sub>6</sub>H<sub>6</sub>, m.p. about 133°.

**Methopicate**: yellow needles from H<sub>2</sub>O. M.p. 164·5° (169·5°).

**Ethochloride**: plates + H<sub>2</sub>O. M.p. 92·5°, anhyd. 122°.

**Ethobromide**: plates + H<sub>2</sub>O from H<sub>2</sub>O or EtOH. M.p. 80°. Loses H<sub>2</sub>O at 100°. Sol. CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O.

**Ethiodide**: yellow prisms from EtOH. M.p. 158° (156–7°). Sol. H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O.

**Propyl chloride**: plates + H<sub>2</sub>O from H<sub>2</sub>O, m.p. 95°. Loses H<sub>2</sub>O at 130–5° and melts at 135°. The anhyd. salt is hygroscopic. Cryst. from CHCl<sub>3</sub> in prisms + CHCl<sub>3</sub>, m.p. 79°.

**Propyl bromide**: hygroscopic cryst. from EtOH, m.p. 148°; plates + 2H<sub>2</sub>O from H<sub>2</sub>O or EtOH, m.p. 66°.

**Propyl iodide**: yellow cryst. from H<sub>2</sub>O or EtOH, m.p. 145°; yellow prisms + CHCl<sub>3</sub> from CHCl<sub>3</sub>.

**Isopropyl iodide**: needles from Me<sub>2</sub>CO. M.p. 136°.

**Butyl iodide**: yellow cryst. from EtOH or prisms from Me<sub>2</sub>CO. M.p. 174°.

**Isobutyl iodide**: yellow cryst. from EtOH or prisms from Me<sub>2</sub>CO. M.p. 161°.

**Isamyl bromide**: yellow needles + H<sub>2</sub>O from EtOH. M.p. 87°, anhyd. 140°.

**Isamyl iodide**: yellowish-green cryst. M.p. 184–5°.

**Hexadecyl iodide**: m.p. 101°.

**Allyl iodide**: cryst. from H<sub>2</sub>O or EtOH. M.p. 177·5°.

**Benzyl chloride**: plates + 3H<sub>2</sub>O from H<sub>2</sub>O. M.p. 65°, anhyd. 170°. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O. KMnO<sub>4</sub> → 2-benzylamino-benzoic acid.

**Benzyl iodide**: cryst. M.p. 135°.

**N-Oxide**: needles + 2H<sub>2</sub>O. M.p. 62°.

**Picrate**: m.p. 143°.

Skraup, *Monatsh.*, 1880, **1**, 316; 1881, **2**, 141.

Walter, *J. prakt. Chem.*, 1894, **49**, 549.

Kneuppel, *Ber.*, 1896, **29**, 704.

Hantzsch, *Ber.*, 1909, **42**, 80.

Barnett, *Chem. News*, 1920, **121**, 205.

König, *Ber.*, 1923, **56**, 1853.

Cohn, Gustavson, *J. Am. Chem. Soc.*, 1928, **50**, 2709.

Cohn, *J. Am. Chem. Soc.*, 1930, **52**, 3685.

Clarke, Davis, *Organic Syntheses*, Collective Vol. I, 466.

Kirkhof, Fedotov, *Chem. Abstracts*, 1933, **27**, 5331.

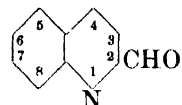
Darzens, Delaby, Hiron, *Bull. soc. chim.*, 1930, **47**, 227.

Kirchhof, Sassosow, *Chem. Zentr.*, 1935, **I**, 2371.

Mikhailov, *Chem. Abstracts*, 1934, **28**, 3736.

Dehn, Cope, *J. Am. Chem. Soc.*, 1926, **48**, 2634.

### Quinoline-2-aldehyde (2-Aldehydoquinoline)



C<sub>10</sub>H<sub>7</sub>ON

MW, 157

Prisms or plates from pet. ether, m.p. 71° (70–1°, 67–9°); needles + ?H<sub>2</sub>O from H<sub>2</sub>O, m.p. 51°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, pet ether. Reduces NH<sub>3</sub>.AgNO<sub>3</sub>.

**Oxime**: needles. M.p. 189° (184°).

**Phenylhydrazone**: yellow plates from EtOH. M.p. 204° (195–8°). Spar. sol. cold EtOH. Prac. insol. H<sub>2</sub>O, Et<sub>2</sub>O.

**p-Nitrophenylhydrazone**: yellow cryst. Sublimes at 225°. M.p. 250°.

Einhorn, *Ber.*, 1886, **19**, 908.

v. Miller, Spady, *Ber.*, 1885, **18**, 3404; 1886, **19**, 132.

Hammick, *J. Chem. Soc.*, 1926, 1303.

Monti, *Chem. Abstracts*, 1934, **28**, 4733.

Cooper, Cohen, *J. Chem. Soc.*, 1932, 723.

Pfitzinger, *J. prakt. Chem.*, 1902, **66**, 264.

Kaufmann, Valette, *Ber.*, 1913, **46**, 57.

Kwartler, Lindwall, *J. Am. Chem. Soc.*, 1937, **59**, 524.

### Quinoline-4-aldehyde (4-Aldehydoquinoline)

Plates + 1H<sub>2</sub>O, m.p. 84–84·5°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, toluene. Anhyd. needles from toluene, m.p. 51–3°. Sol. Et<sub>2</sub>O, toluene, xylene. Reduces Tollen's.

**Oxime**: needles from MeOH. M.p. 181–2°.

**p-Nitrophenylhydrazone**: yellow prisms from EtOH. M.p. 261–2°.

See last reference above.

### Quinoline-6-aldehyde (6-Aldehydoquinoline)

Needles + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 55°, anhyd. 75–6°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: reddish-yellow needles. M.p. 244°.

**Anil**: needles from dil. EtOH. M.p. 99°.

**Oxime**: yellow needles from EtOH. M.p. 191°.

*Phenylhydrazone*: reddish-yellow needles +  $\text{H}_2\text{O}$  from EtOH. M.p.  $185^\circ$ .

*Semicarbazone*: yellow needles from EtOH. M.p.  $239^\circ$ .

*Azine*: yellow needles from EtOH. M.p.  $261^\circ$ . Spar. sol. EtOH.

*Methiodide*: yellow cryst. from EtOH. M.p.  $218^\circ$ . Sol.  $\text{H}_2\text{O}$ .

Howitz, Philipp, *Ann.*, 1913, **396**, 28.

**Quinoline-8-aldehyde** (8-Aldehydoquinoline).

Needles from dil. EtOH. M.p.  $94-5^\circ$ . Sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Volatile in steam.

*B,HCl*: cryst. from EtOH. M.p.  $213^\circ$ . Sol.  $\text{H}_2\text{O}$ , EtOH.

*B,HI*: red cryst. from  $\text{H}_2\text{O}$ . M.p.  $228^\circ$  decomp.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: reddish-yellow cryst. from alc. HCl. M.p.  $250^\circ$  decomp.

*Anil*: yellow cryst. from EtOH. M.p.  $82^\circ$ .

*Oxime*: plates +  $\frac{1}{2}\text{H}_2\text{O}$  from dil. EtOH. M.p.  $115^\circ$ .

*Phenylhydrazone*: yellow needles from EtOH. M.p.  $176^\circ$ .

*Semicarbazone*: needles from  $\text{C}_6\text{H}_6$ . M.p.  $238-9^\circ$ .

*Azine*: yellow needles from  $\text{C}_6\text{H}_6$ . M.p.  $248-9^\circ$ . Spar. sol.  $\text{C}_6\text{H}_6$ .

Howitz, *Ber.*, 1902, **35**, 1274.

Howitz, Schwenk, *Ber.*, 1905, **38**, 1289.

Howitz, Köpke, *Ann.*, 1913, **396**, 39.

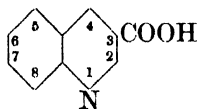
## 2-Quinoline-carbinol.

See 2-Hydroxymethyl-quinoline.

## Quinoline-2-carboxylic Acid.

See Quinaldinic Acid.

## Quinoline-3-carboxylic Acid



$\text{C}_{10}\text{H}_7\text{O}_2\text{N}$

MW, 173

Plates from dil. EtOH. M.p.  $275^\circ$  part. decomp. ( $271-2^\circ$ ). Sol. hot  $\text{H}_2\text{O}$ , EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ . Alk.  $\text{KMnO}_4 \rightarrow$  pyridine-2 : 3 : 5-tricarboxylic acid. Heat with lime  $\rightarrow$  quinoline.

*Me ester*:  $\text{C}_{11}\text{H}_9\text{O}_2\text{N}$ . MW, 187. M.p.  $76^\circ$ . *Picrate*: m.p.  $187-8^\circ$ .

*Et ester*:  $\text{C}_{12}\text{H}_{11}\text{O}_2\text{N}$ . MW, 201. M.p.  $65^\circ$ .

*Nitrile*: 3-cyanoquinoline.  $\text{C}_{10}\text{H}_6\text{N}_2$ . MW, 154. M.p.  $108^\circ$ .

*Amide*:  $\text{C}_{10}\text{H}_8\text{ON}_2$ . MW, 172. Needles from  $\text{H}_2\text{O}$ . M.p.  $198-9^\circ$  ( $195^\circ$ ). Mod. sol. EtOH.

*Picrate*: needles. M.p.  $217-18^\circ$ . Spar. sol. cold EtOH.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-yellow plates or needles. Sol. hot  $\text{H}_2\text{O}$ .

Doebner, v. Miller, *Ber.*, 1885, **18**, 1644.

Riedel, *Ber.*, 1883, **16**, 1613.

Graebe, Caro, *Ber.*, 1880, **13**, 101.

Mills, Watson, *J. Chem. Soc.*, 1910, **97**, 745.

Koller, Ruppertsberg, Strang, *Monatsh.*, 1929, **52**, 66.

## Quinoline-4-carboxylic Acid.

See Cinchoninic Acid.

## Quinoline-5-carboxylic Acid.

Cryst. from hot AcOH. M.p.  $338-40^\circ$ . Spar. sol. EtOH,  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ . Sublimes. Sol. dil. acids and alkalis. Heat with lime  $\rightarrow$  quinoline.

*Nitrile*: 5-cyanoquinoline.  $\text{C}_{10}\text{H}_6\text{N}_2$ . MW, 154. Needles from ligroin, m.p.  $89^\circ$  ( $87-8^\circ$ ): needles +  $1\frac{1}{2}\text{H}_2\text{O}$  from dil. EtOH, m.p.  $70^\circ$ . Does not boil below  $360^\circ$ . Sol. EtOH,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ . Spar. sol.  $\text{H}_2\text{O}$ , ligroin.

*B,HCl*: needles or prisms +  $\text{H}_2\text{O}$ . Sol. EtOH.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow plates or needles. Spar. sol.  $\text{H}_2\text{O}$ .

Bedall, Fischer, *Ber.*, 1881, **14**, 2574; 1882, **15**, 683.

Jakubowski, *Ber.*, 1910, **43**, 3026.

Schlosser, Skraup, *Monatsh.*, 1881, **2**, 519.

Lellmann, Alt, *Ann.*, 1887, **237**, 318.

See also Skraup, Brunner, *Monatsh.*, 1886, **7**, 153, 519.

## Quinoline-6-carboxylic Acid.

Prisms, plates or needles. M.p.  $291-2^\circ$  ( $290-1^\circ$ ). Sol. warm EtOH. Spar. sol.  $\text{H}_2\text{O}$ . Sublimes. Sol. dil. acids and alkalis.

*Et ester*:  $\text{C}_{12}\text{H}_{11}\text{O}_2\text{N}$ . MW, 201. Needles from dil. EtOH. M.p.  $50^\circ$ . *B,HCl*: needles from EtOH. M.p.  $210^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ .

*Amide*:  $\text{C}_{10}\text{H}_8\text{ON}_2$ . MW, 172. Yellow plates from  $\text{C}_6\text{H}_6$ -EtOH. M.p.  $174^\circ$ . *Ethiodide*: red or greenish-yellow cryst. from dil. EtOH. M.p.  $229^\circ$  part. decomp.

*Nitrile*: cryst. from  $\text{C}_6\text{H}_6$ -ligroin. M.p.  $131^\circ$  ( $135^\circ$ ). Sublimes. Sol. dil. HCl with red col.

*B,HCl*: needles +  $\text{H}_2\text{O}$ . Hyd. by  $\text{H}_2\text{O}$ .

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: reddish-yellow plates.

Schlosser, Skraup, *Monatsh.*, 1881, **2**, 526.

v. Georgievics, *Monatsh.*, 1891, **12**, 306.

Biedermann, *Ber.*, 1889, **22**, 2762.

Einhorn, Feibelman, *Ber.*, 1909, **42**, 4854.

Howitz, Philipp, *Ann.*, 1913, **396**, 29.

I.G., F.P., 727,528, (*Chem. Abstracts*, 1932, **26**, 5104).

**Quinoline-7-carboxylic Acid.**

Needles from  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . M.p.  $248-9^\circ$  ( $248.5-250^\circ$ ,  $247^\circ$ ). Sol.  $\text{EtOH}$ . Spar. sol. hot  $\text{H}_2\text{O}$ . Prac. insol. cold  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ . Sublimes. Heat with lime  $\rightarrow$  quinoline.

$B, HCl$ : prisms +  $\text{H}_2\text{O}$  from  $\text{HCl.Aq}$ .  
 $B_2, H_2PtCl_6$ : orange prisms or needles.

Skraup, Brunner, *Monatsh.*, 1886, 7, 142.  
 Fischer, van Loo, *Ber.*, 1886, 19, 2473.

**Quinoline-8-carboxylic Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $187^\circ$  ( $186-187.5^\circ$ ). Mod. sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Sol. dil. acids and alkalis. Sublimes.  $\text{KMnO}_4 \rightarrow$  quinolinic acid.

*Nitrile*: 8-cyanoquinoline. Needles from 50%  $\text{EtOH}$ . M.p.  $84^\circ$ .  $B_2, H_2PtCl_6$ : orange-yellow needles from  $\text{H}_2\text{O}$ .

$B, HCl$ : prisms from dil.  $\text{HCl}$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol. dil.  $\text{EtOH}$ .

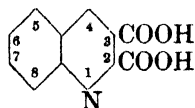
$B_2, H_2PtCl_6$ : orange-yellow needles or red granular cryst. from dil.  $\text{HCl}$ . Spar. sol.  $\text{H}_2\text{O}$ .

Schlosser, Skraup, *Monatsh.*, 1881, 2, 530.  
 Howitz, *Ber.*, 1902, 35, 1275.

Bedall, Fischer, *Ber.*, 1882, 15, 683.

Lellmann, Reusch, *Ber.*, 1889, 22, 1391.

Chakravarti, Granapati, *Chem. Abstracts*, 1935, 29, 1090.

**Quinoline-2 : 3-dicarboxylic Acid (Acridinic acid)**

$\text{C}_{11}\text{H}_7\text{O}_4\text{N}$  MW, 217

Needles + 2 or  $3\text{H}_2\text{O}$  from hot  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . Starts to lose  $\text{CO}_2$  at  $105^\circ$  leaving residue, m.p.  $274^\circ$ . Sol.  $\text{EtOH}$ . Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Decomp. at  $120^\circ \rightarrow$  quinoline-3-carboxylic acid. Does not form salts with acids. Heat with lime  $\rightarrow$  quinoline.  $\text{FeSO}_4 \rightarrow$  reddish-yellow col.

*Me ester*:  $\text{C}_{12}\text{H}_9\text{O}_4\text{N}$ . MW, 231. M.p.  $174-6^\circ$ .

*Di-Me ester*:  $\text{C}_{13}\text{H}_{11}\text{O}_4\text{N}$ . MW, 245. Prisms from  $\text{MeOH}$  or  $\text{C}_6\text{H}_6$ . M.p.  $107-8^\circ$ .

*Et ester*:  $\text{C}_{13}\text{H}_{11}\text{O}_4\text{N}$ . MW, 245. M.p.  $170-2^\circ$ .

*Di-Et ester*:  $\text{C}_{15}\text{H}_{15}\text{O}_4\text{N}$ . MW, 273. Prisms from dil.  $\text{EtOH}$ . M.p.  $55-6^\circ$ .

*Anhydride*:  $\text{C}_{11}\text{H}_5\text{O}_3\text{N}$ . MW, 199. M.p.  $225^\circ$  ( $223^\circ$ ).

*Amide*:  $\text{C}_{11}\text{H}_8\text{O}_3\text{N}_2$ . MW, 216. Cryst. for  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . M.p.  $174-6^\circ$  decomp.

*Diamide*:  $\text{C}_{11}\text{H}_9\text{O}_2\text{N}_3$ . MW, 215. Sinters at  $250-70^\circ \rightarrow$  imide.

*Anilide*: m.p.  $187^\circ$  decomp.

*Dianilide*: sinters at  $245^\circ$ .

*Imide*:  $\text{C}_{11}\text{H}_6\text{O}_2\text{N}_2$ . MW, 198. M.p.  $316^\circ$  ( $314-15^\circ$ ).

*Phenylimide*: m.p.  $319-20^\circ$ .

Graebe, Caro, *Ber.*, 1880, 13, 100.

Konopnicki, Sucharda, *Chem. Abstracts*, 1928, 22, 785.

Koller, Strang, *Monatsh.*, 1928, 50, 48.

Hozer, v. Niementowski, *J. prakt. Chem.*, 1927, 116, 43.

**Quinoline-2 : 4-dicarboxylic Acid.**

Needles from  $\text{H}_2\text{O}$ . M.p.  $246^\circ$  decomp. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether. Heat at  $240^\circ \rightarrow$  cinchoninic acid. Heat with lime  $\rightarrow$  quinoline.

*2-Nitrile*: 2-cyanocinchoninic acid.  $\text{C}_{11}\text{H}_6\text{O}_2\text{N}_2$ . MW, 198. Needles. M.p.  $226^\circ$ .

Pfützing, *J. prakt. Chem.*, 1897, 56, 308; 1902, 66, 264.

Doebner, Peters, *Ber.*, 1889, 22, 3009.

**Quinoline-2 : 6-dicarboxylic Acid.**

Cryst. M.p.  $275-80^\circ$  decomp.

v. Miller, *Ber.*, 1890, 23, 2261.

**Quinoline-5 : 6-dicarboxylic Acid.**

Plates +  $\text{H}_2\text{O}$  from dil.  $\text{HCl}$ . M.p.  $238-41^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .

$B, HCl$ : prisms for conc.  $\text{HCl}$ . Hyd. by  $\text{H}_2\text{O}$ .

$B_2, H_2PtCl_6$ : brownish-yellow needles. Decomp. above  $240^\circ$ .

Hepner, *Monatsh.*, 1906, 27, 1062.

**Quinoline-5 : 8-dicarboxylic Acid.**

Needles +  $2\text{H}_2\text{O}$  from dil.  $\text{HCl}$ . M.p.  $268-70^\circ$ . Prac. insol. cold  $\text{H}_2\text{O}$ . At  $270-80^\circ \rightarrow$  quinoline-5- and -8-carboxylic acids.

$B, HCl$ : cryst. +  $1\frac{1}{2}\text{H}_2\text{O}$ . Hyd. by  $\text{H}_2\text{O}$ .

$B_2, H_2PtCl_6$ : yellowish-red cryst. Decomp. by  $\text{H}_2\text{O}$  or  $\text{EtOH}$ .

Skraup, Brunner, *Monatsh.*, 1886, 7, 149.

**Quinoline-7 : 8-dicarboxylic Acid.**

Prisms or needles +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $206-7^\circ$  decomp. Spar. sol. hot  $\text{EtOH}$ , cold  $\text{H}_2\text{O}$ . Part. decomp. on steam dist.

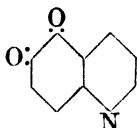
$B, HCl$ : prisms. M.p.  $212^\circ$  decomp. Hyd. by  $\text{H}_2\text{O}$ .

Haid, *Monatsh.*, 1906, 27, 333.

**Quinoline-hydroquinone.**

See 5 : 8-Dihydroxyquinoline.

## Quinoline-5 : 6-quinone

 $C_9H_5O_2N$ 

MW, 159

Prisms. Does not melt below  $350^\circ$ . Sol. AcOH and min. acids. Prac. insol.  $H_2O$  and most org. solvents.

5 - Oxime : 5-nitroso-6-hydroxyquinoline. Golden-yellow needles from EtOH or AcOH. Chars above  $180^\circ$  without melting. Sol. dil. acids and alkalis. Spar. sol.  $Et_2O$ . Insol.  $H_2O$ .  $HNO_3 \rightarrow$  5-nitro-6-hydroxyquinoline.  $SnCl_2 \rightarrow$  5-amino-6-hydroxyquinoline.

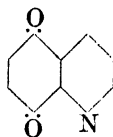
Dioxime : needles from EtOH. Decomp. at  $190^\circ$ .

Mathëus, *Ber.*, 1888, **21**, 1887.

v. Kostanecki, *Ber.*, 1891, **24**, 150.

v. Kostanecki, Reicher, *ibid.*, 158.

## Quinoline-5 : 8-quinone

 $C_9H_5O_2N$ 

MW, 159

Greenish needles from EtOH. Decomp. at  $110-20^\circ$ . Very unstable towards alkalis.  $SO_2 \rightarrow$  5 : 8-dihydroxyquinoline.

5 - Oxime : 5-nitroso-8-hydroxyquinoline. Needles from EtOH. Darkens at  $220^\circ$ . Decomp. at  $245^\circ$ . Prac. insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ . Cold  $HNO_3$  or  $K_3Fe(CN)_6 \rightarrow$  5-nitro-8-hydroxyquinoline. Hot  $HNO_3 \rightarrow$  5 : 7-dinitro-8-hydroxyquinoline.  $SnCl_2 \rightarrow$  5-amino-8-hydroxyquinoline.

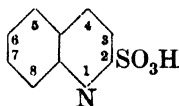
Dioxime : cryst. from dil. EtOH. Decomp. above  $200^\circ$ . Sol. alkalis with yellow col.

Fischer, Renouf, *Ber.*, 1884, **17**, 1644.

v. Kostanecki, *Ber.*, 1891, **24**, 152.

v. Kostanecki, Reicher, *ibid.*, 157.

## Quinoline-2-sulphonic Acid

 $C_9H_7O_3NS$ 

MW, 209

Needles from dil. HCl. Does not melt below  $270^\circ$ . Spar. sol. cold  $H_2O$ . Boiling  $H_2O \rightarrow$  carbostyryl +  $SO_2$ . Alkali salts sol.  $H_2O$ .

Besthorn, Geisselbrecht, *Ber.*, 1920, **53**, 1021.

## Quinoline-3-sulphonic Acid.

Needles +  $H_2O$  from  $H_2O$ . Loses  $H_2O$  at  $110^\circ$ . Does not melt below  $270^\circ$ . Mod. sol. hot  $H_2O$ .

Ba salt : needles +  $H_2O$  from  $H_2O$ . Mod. sol.  $H_2O$ .

Besthorn, Geisselbrecht, *Ber.*, 1920, **53**, 1032.

## Quinoline-4-sulphonic Acid.

Needles from dil. HCl. Does not melt below  $270^\circ$ . Alkali salts sol.  $H_2O$ .

Ca salt : needles +  $4H_2O$ . Sol. cold  $H_2O$ .

Ag salt : needles. Sol.  $H_2O$ .

Besthorn, Geisselbrecht, *Ber.*, 1920, **53**, 1023.

## Quinoline-5-sulphonic Acid.

Cryst. +  $H_2O$ . Sol.  $H_2O$ .  $Sn + HCl \rightarrow$  1 : 2 : 3 : 4-tetrahydroquinoline-5-sulphonic acid. Br in AcOH  $\rightarrow$  3-bromoquinoline-5-sulphonic acid. Br  $\rightarrow$  3 : 5 : 8-tribromoquinoline + 3 : 5 : 6 : 8-tetrabromoquinoline. Heat with  $H_2SO_4$  at  $250-300^\circ \rightarrow$  quinoline-6-sulphonic acid. KOH fusion  $\rightarrow$  5-hydroxyquinoline.

Ca salt : needles +  $5H_2O$  from  $H_2O$  or dil. EtOH.

Bedall, Fischer, *Ber.*, 1882, **15**, 684.

Fischer, *Ber.*, 1882, **15**, 1979; 1887, **20**, 731.

Claus, *J. prakt. Chem.*, 1888, **37**, 258.

La Coste, Valeur, *Ber.*, 1887, **20**, 95.

Riemerschmied, *Ber.*, 1883, **16**, 721.

## Quinoline-6-sulphonic Acid.

Needles +  $1\frac{1}{2}H_2O$ . Does not melt below  $260^\circ$ . Spar. sol. cold  $H_2O$ , cold EtOH. Br  $\rightarrow$  3 : 6-dibromoquinoline + 3 : 6 : 8-tribromoquinoline. KOH fusion  $\rightarrow$  6-hydroxyquinoline. KCN fusion  $\rightarrow$  6-cyanoquinoline.

Happ, *Ber.*, 1884, **17**, 192.

Fischer, Willmack, *Ber.*, 1884, **17**, 440.

Knueppel, *Ber.*, 1896, **29**, 707.

v. Georgievics, *Monatsh.*, 1887, **8**, 577.

## Quinoline-7-sulphonic Acid.

Needles from  $H_2O$ . Decomp. above  $300^\circ$ . Sol.  $H_2O$ . Spar. sol. EtOH.

Na salt : cryst. +  $3H_2O$ . Sol.  $H_2O$ .

K salt : cryst. +  $1\frac{1}{2} \cdot 2H_2O$ .

Ca salt : needles +  $4H_2O$ . Sol.  $H_2O$ .

Ba salt : cryst. +  $4H_2O$ .

Pb salt : cryst. powder. Sol.  $H_2O$ .

Chloride :  $C_9H_6O_3NClS$ . MW, 227.5. Viscous brown mass. Prac. insol.  $Et_2O$ ,  $CHCl_3$ .

Amide :  $C_9H_7O_3N_2S$ . MW, 208. Cryst. M.p.  $119^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

Claus, *J. prakt. Chem.*, 1888, **37**, 261.



**Quinoline-8-sulphonic Acid.**

Prisms. Spar. sol.  $\text{H}_2\text{O}$ . Dist.  $\rightarrow$  3:7'- or 4:7'-diquinolyl.  $\text{KMnO}_4 \rightarrow$  quinolinic acid + 2-amino-3-sulphobenzoic acid.  $\text{Sn} + \text{HCl} \rightarrow$  1:2:3:4-tetrahydroquinoline-8-sulphonic acid.  $\text{Br} \rightarrow$  3:6:8-tribromoquinoline + 3:5:6:8-tetrabromoquinoline.  $\text{KOH}$  fusion  $\rightarrow$  8-hydroxyquinoline.  $\text{H}_2\text{SO}_4$  at  $300^\circ \rightarrow$  quinoline-6-sulphonic acid. Fuming  $\text{HNO}_3$  at  $160^\circ \rightarrow$  8-nitroquinoline.  $\text{KCN}$  fusion  $\rightarrow$  8-cyano- + 5-cyano-quinolines.

$\text{NH}_4$  salt: plates +  $\text{H}_2\text{O}$ .

$\text{Na}$  salt: needles +  $5\text{H}_2\text{O}$ .

$\text{K}$  salt: prisms +  $2\text{H}_2\text{O}$ .

$\text{Ca}$  salt: needles +  $6\text{H}_2\text{O}$ .

$\text{Ba}$  salt: plates +  $9\text{H}_2\text{O}$ .

$\text{Pb}$  salt: prisms. Spar. sol.  $\text{H}_2\text{O}$ .

$\text{Me}$  ester:  $\text{C}_{10}\text{H}_9\text{O}_3\text{NS}$ . MW, 223. Prisms. M.p.  $96^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ . Prac. insol. ligroin.

$\text{Et}$  ester:  $\text{C}_{11}\text{H}_{11}\text{O}_3\text{NS}$ . MW, 237. Cryst. M.p.  $73^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

$\text{Benzyl}$  ester:  $\text{C}_{16}\text{H}_{13}\text{O}_3\text{NS}$ . MW, 299. Plates or prisms from  $\text{Et}_2\text{O}$ . M.p.  $84^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol. ligroin.

$\text{Chloride}$ : needles. M.p.  $124^\circ$  ( $122^\circ$ ). Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

$\text{Amide}$ : needles from  $\text{H}_2\text{O}$ . M.p.  $183-4^\circ$ .

Bedall, Fischer, *Ber.*, 1882, **15**, 684.

Fischer, *Ber.*, 1882, **15**, 1979; 1887, **20**, 731.

La Coste, Valeur, *Ber.*, 1887, **20**, 95.

Claus, *J. prakt. Chem.*, 1888, **37**, 258.

v. Georgievics, *Monatsh.*, 1887, **8**, 641.

Claus, Steinitz, *Ann.*, 1894, **282**, 132.

Claus, Küttner, *Ber.*, 1886, **19**, 925.

Hoogewerff, van Dorp, *Rec. trav. chim.*, 1889, **8**, 184.

**Quinolinic Acid** (*Pyridine-2:3-dicarboxylic acid*)



$\text{C}_7\text{H}_5\text{O}_4\text{N}$

MW, 167

Prisms from  $\text{H}_2\text{O}$ . M.p.  $190-5^\circ$  decomp. (rapid heat); decomp. at  $110^\circ$  (slow heat). Sol. 183 parts  $\text{H}_2\text{O}$  at  $6.5^\circ$ . Prac. insol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Heat  $\rightarrow$  nicotinic acid.

$1\text{-Me}$  ester:  $\text{C}_8\text{H}_7\text{O}_4\text{N}$ . MW, 181. Prisms +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $90^\circ$ . Obtained anhyd. from  $\text{AcOEt}$ , m.p.  $123^\circ$ . Sol. hot  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

$2\text{-Me}$  ester: cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $106^\circ$ . Sol.  $\text{H}_2\text{O}$  and most org. solvents.

$1\text{-Me}$  ester  $2\text{-chloride}$ :  $\text{C}_8\text{H}_6\text{O}_3\text{NCl}$ . MW, 199.5. Needles. M.p.  $126^\circ$ .

$\text{Di-Me}$  ester:  $\text{C}_9\text{H}_9\text{O}_4\text{N}$ . MW, 195. Plates from  $\text{CS}_2$ -ligroin. M.p.  $54-5^\circ$ .

$1\text{-Et}$  ester:  $\text{C}_9\text{H}_9\text{O}_4\text{N}$ . MW, 195. Plates from  $\text{C}_6\text{H}_6$ . M.p.  $132^\circ$ . Also prisms +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .

$1\text{-Et}$  ester  $2\text{-chloride}$ :  $\text{C}_9\text{H}_8\text{O}_3\text{NCl}$ . MW, 213.5. M.p.  $163^\circ$ .

$1\text{-Me}$   $2\text{-Et}$  ester:  $\text{C}_{10}\text{H}_{11}\text{O}_4\text{N}$ . MW, 209. B.p.  $250-5^\circ$  decomp.  $\text{B}_2\text{H}_2\text{PtCl}_6$ : orange-red cryst. from alc.  $\text{HCl}$ . M.p.  $165^\circ$  decomp.

$2\text{-Me}$   $1\text{-Et}$  ester: b.p.  $254-8^\circ$  decomp.  $\text{B}_2\text{H}_2\text{PtCl}_6$ : yellow needles from alc.  $\text{HCl}$ . M.p.  $174^\circ$  decomp.

$\text{Di-Et}$  ester:  $\text{C}_{11}\text{H}_{13}\text{O}_4\text{N}$ . MW, 223. B.p.  $280-5^\circ$  part. decomp.

$\text{Dichloride}$ :  $\text{C}_7\text{H}_3\text{O}_2\text{NCl}_2$ . MW, 204. B.p.  $159^\circ/19$  mm.

$1\text{-Amide}$ :  $\text{C}_7\text{H}_6\text{O}_3\text{N}_2$ . MW, 166. Prisms. M.p.  $168.5^\circ$  decomp. Spar. sol. cold  $\text{H}_2\text{O}$ .

$2\text{-Amide}$ : prisms. M.p.  $160^\circ$  decomp.

$\text{Diamide}$ :  $\text{C}_7\text{H}_4\text{O}_2\text{N}_3$ . MW, 165. Needles from  $\text{EtOH}$ . M.p.  $209^\circ$  decomp. ( $190^\circ$  decomp.). Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

$\text{Imide}$ :  $\text{C}_7\text{H}_4\text{O}_2\text{N}_2$ . MW, 148. Needles from  $\text{EtOH}$ . M.p.  $233^\circ$  ( $230^\circ$ ). Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{AcOH}$ . Insol.  $\text{C}_6\text{H}_6$ , ligroin.

$\text{Anhydride}$ :  $\text{C}_7\text{H}_3\text{O}_3\text{N}$ . MW, 149. M.p.  $161-2^\circ$ .

$3\text{-Nitrile}$ : 3-cyanopicolinic acid.  $\text{C}_7\text{H}_4\text{O}_2\text{N}_2$ . MW, 148. Needles from  $\text{H}_2\text{O}$ . M.p.  $175-6^\circ$ . Sol.  $\text{EtOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , pet. ether.

$\text{Dihydrazide}$ : needles from  $\text{EtOH}$ . M.p.  $224^\circ$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ .

$\text{Me-betaine}$ : plates +  $\text{H}_2\text{O}$ . M.p.  $151^\circ$  decomp. Sol. warm  $\text{H}_2\text{O}$ .

Hoogewerff, van Dorp, *Ber.*, 1879, **12**, 747; *Ann.*, 1880, **204**, 87, 116.

Phillips, *Ann.*, 1895, **288**, 255.

Scheiber, Knothe, *Ber.*, 1912, **45**, 2256.

Kirpal, *Monatsh.*, 1900, **21**, 959; 1901, **22**, 361.

Meyer, *Monatsh.*, 1901, **22**, 580.

Engler, *Ber.*, 1894, **27**, 1787.

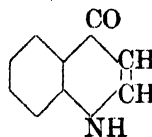
Stix, Bulgatsch, *Ber.*, 1932, **65**, 11.

Sucharda, *Ber.*, 1925, **58**, 1727.

 **$\alpha$ -Quinolone.**

See Carbostyryl.

**$\gamma$ -Quinolone** (*Keto form of 4-hydroxyquinoline*)



$\text{C}_9\text{H}_7\text{ON}$

MW, 145

Cryst. from EtOH. M.p. 235°.

Acetyl: m.p. 228°.

Phenylhydrazone: needles from dil. EtOH. M.p. 168°.

N-Me: see Echinopsine.

N-Et: dark resin. Sol. H<sub>2</sub>O. Prac. insol. Et<sub>2</sub>O. Chloroaurate: m.p. 155° decomp.

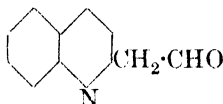
Reissert, *Ber.*, 1887, **20**, 3109; 1888, **21**, 1376.

Meyer, *Monatsh.*, 1906, **27**, 265.

**$\gamma$ -Quinolone-2-carboxylic Acid (Keto form of kynurenic acid).**

The compound (m.p. 167°) regarded as  $\gamma$ -quinolone-2-carboxylic acid by Heller and Sourlis (*Ber.*, 1908, **41**, 2699) has been shown by Meisenheimer and Stotz (*Ber.*, 1925, **58**, 2334) to be quinaldinic acid N-oxide.

**2-Quinolylacetaldehyde**



C<sub>11</sub>H<sub>9</sub>ON

MW, 171

Cryst. from H<sub>2</sub>O or EtOH. M.p. 184° (103-4°). Sol. acids. Reduces NH<sub>3</sub>.AgNO<sub>3</sub>.

Oxime: m.p. 205°.

Phenylhydrazone: yellow needles from EtOH. M.p. 198-9°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: yellow cryst. + 2H<sub>2</sub>O from dil. HCl.

Semicarbazone: needles from EtOH. M.p. 244° decomp.

Picrate: yellow cryst. from dil. EtOH. M.p. 212°.

Einhorn, *Ber.*, 1886, **19**, 908.

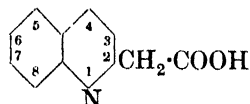
Einhorn, Sherman, *Ann.*, 1895, **287**, 38.

Hupe, Schramme, *Z. physiol. Chem.*, 1928, **177**, 315.

Kenner, Nandi, *Ber.*, 1936, **69**, 639.

Borsche, Manteuffel, *Ann.*, 1936, **526**, 45.

**2-Quinolylacetic Acid**



C<sub>11</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 187

Prisms from MeOH. M.p. 274-5° (271-2°). Sublimes. Ca salt insol. H<sub>2</sub>O, EtOH.

Me ester: C<sub>12</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 201. Flakes from ligroin. M.p. 72°.

Et ester: C<sub>13</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 215. Plates from ligroin. M.p. 68-9° (67°). B.p. 240°/16 mm. Picrate: m.p. 152°.

Nitrile: C<sub>11</sub>H<sub>10</sub>ON<sub>2</sub>. MW, 186. M.p. 53-4°. B.p. 140°/1 mm. Picrate: m.p. 176-7° decomp.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: dimorphous. Needles and brownish-red plates. Sol. H<sub>2</sub>O.

Picrate: yellow needles from MeOH. M.p. 236-7°.

Einhorn, Sherman, *Ann.*, 1895, **287**, 40.

Kenner, Nandi, *Ber.*, 1936, **69**, 639.

Borsche, Manteuffel, *Ann.*, 1936, **526**, 22.

**8-Quinolylacetic Acid.**

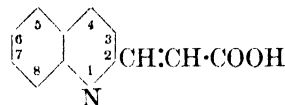
Free acid not described.

Nitrile: C<sub>11</sub>H<sub>8</sub>N<sub>2</sub>. MW, 168. Cryst. M.p. 88°.

Claus, D.R.P., 98,272, (*Chem. Zentr.*, 1898, **II**, 744).

Howitz, Nöther, *Ber.*, 1906, **39**, 2706.

**2-[2-Quinolyl]-acrylic Acid**



C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>N

MW, 199

Plates from MeOH. Decomp. at 194-6° (190-5°). KMnO<sub>4</sub> (according to conditions)  $\rightarrow$  either quinoline-2-aldehyde or 1:2-dihydroxy-2-[2-quinolyl]-propionic acid. SnCl<sub>2</sub> or NaHg  $\rightarrow$  2-[2-quinolyl]-propionic acid. Na + EtOH or Sn + HCl  $\rightarrow$  2-[2-(1:2:3:4-tetrahydroquinolyl)]-propionic acid.

Et ester: C<sub>14</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 227. Needles from ligroin. M.p. 73°.

Amide: C<sub>12</sub>H<sub>10</sub>ON<sub>2</sub>. MW, 198. Needles from H<sub>2</sub>O. M.p. 175-6°. Sol. warm. EtOH, C<sub>6</sub>H<sub>6</sub>.

B.HCl: m.p. 216-18°.

B.HBr: m.p. 218-20°.

Acetate: m.p. 203°.

Einhorn, *Ber.*, 1886, **19**, 908.

Einhorn, Sherman, *Ann.*, 1895, **287**, 27.

v. Miller, Spady, *Ber.*, 1885, **18**, 3403; 1886, **19**, 130.

Alberts, Bachmann, *J. Am. Chem. Soc.*, 1935, **57**, 1285.

**2-[4-Quinolyl]-acrylic Acid.**

Needles from EtOH-AcOH. M.p. 250-5° decomp. Spar. sol. EtOH. Prac. insol. H<sub>2</sub>O. HI(+ P) in AcOH  $\rightarrow$  2-[4-quinolyl]-propionic acid.

$B_2H_2PtCl_6$ : yellow needles +  $1\frac{1}{2}H_2O$  from  $H_2O$ .

Koenigs, Müller, *Ber.*, 1904, **37**, 1338.

### Quinolyamine.

See Aminoquinoline.

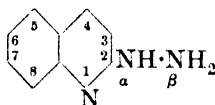
### $\alpha$ -Quinolylicarbinol.

See 2-Hydroxymethyl-quinoline.

### Quinolyethylene.

See Vinylquinoline.

### 2-Quinolyldiazine (2-Hydrazinoquinoline)



$C_9H_9N_3$

MW, 159

Cryst. from  $C_6H_6$ . M.p.  $142-3^\circ$  ( $134-5^\circ$ ). Sol. EtOH. Spar. sol.  $Et_2O$ , ligroin.

$B_2H_2PtCl_6$ : cryst. ppt. M.p.  $170^\circ$  decomp.

Picrate: m.p.  $187^\circ$  decomp.

$\beta$ -N-Phenyl: sym.-phenylquinolyldiazine. Needles from EtOH. M.p.  $191^\circ$ . Unstable. Sol.  $CHCl_3$ , AcOH. Spar. sol. EtOH. Prac. insol.  $Et_2O$ .

$\beta$ -N-Acetyl: prisms from EtOH. M.p.  $195^\circ$ .  $\beta$ -N-Benzoyl: needles from  $Me_2CO$ . M.p.  $204^\circ$ .

Perkin, Robinson, *J. Chem. Soc.*, 1913, **103**, 1978.

Marckwald, Meyer, *Ber.*, 1900, **33**, 1885.

Ephraim, *Ber.*, 1891, **24**, 2818.

Fargher, Furness, *J. Chem. Soc.*, 1915, **107**, 697.

### 5-Quinolyldiazine.

Yellow needles from  $H_2O$ . M.p.  $150-1^\circ$ . Sol. EtOH. Spar. sol.  $C_6H_6$ . Insol. pet. ether.

$B_2HCl$ : yellow needles. M.p.  $248^\circ$ .

Duften, *J. Chem. Soc.*, 1892, **61**, 785.

### 6-Quinolyldiazine.

Non-crystalline. Readily resinifies.

$B_2HCl$ : cryst. from dil. EtOH.

Benzylidene deriv.: yellowish-red needles from  $H_2O$  or red cryst. from EtOH. M.p.  $203^\circ$ .

Knueppel, *Ann.*, 1900, **310**, 82.

### 8-Quinolyldiazine.

Needles. M.p.  $64^\circ$ .

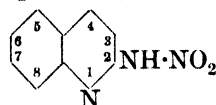
$B_2HCl$ : yellow prisms.

Duften, *J. Chem. Soc.*, 1891, **59**, 757.

### 2-Quinoly Mercaptan.

See Thiocarbostryl.

### 2-Quinolylnitramine (2-Nitraminoquinoline, N-nitro-2-aminoquinoline)



$C_9H_7O_2N_3$

MW, 189

Yellowish needles from AcOH. M.p.  $223-5^\circ$  decomp. Turns red in air.

Tschitschibabin, Witkovsky, Lapschin, *Ber.*, 1925, **58**, 806.

### 4-Quinolylnitramine (4-Nitraminoquinoline, N-nitro-4-aminoquinoline).

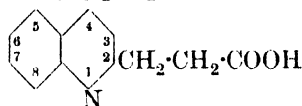
Yellow needles +  $H_2O$  from  $H_2O$ . M.p.  $207^\circ$  decomp. Sol. EtOH, hot  $H_2O$ .

$B_2H_2PtCl_6$ : orange-red cryst. Decomp. at  $210^\circ$ . Hyd. by  $H_2O$ .

See previous reference and also

Claus, Frobenius, *J. prakt. Chem.*, 1897, **56**, 202.

### 2-[2-Quinoly]-propionic Acid



$C_{12}H_{11}O_2N$

MW, 201

Plates from  $C_6H_6$ . M.p.  $122-3^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $Me_2CO$ . Insol.  $H_2O$ , ligroin.

Amide:  $C_{12}H_{12}ON_2$ . MW, 200. Needles from  $C_6H_6$ . M.p.  $149-50^\circ$ . Sol. hot  $H_2O$ , EtOH,  $C_6H_6$ . Insol. pet. ether.

Hydrazide: m.p.  $165^\circ$ . Reduces Fehling's and  $NH_3AgNO_3$ .

$B_2H_2PtCl_6$ : brownish-red plates from dil. HCl. M.p.  $197^\circ$  decomp.

Einhorn, Sherman, *Ann.*, 1895, **287**, 29.

Koenigs, *Ber.*, 1900, **33**, 220.

Kermack, Muir, *J. Chem. Soc.*, 1931, **3092**.

### 2-[4-Quinoly]-propionic Acid.

Needles from  $H_2O$ . M.p.  $202-3^\circ$ . Sol. EtOH, hot  $H_2O$ , boiling  $Me_2CO$ . Na + EtOH  $\rightarrow$  2-[4-(1:2:3:4-tetrahydroquinoly)]-propionic acid.

Koenigs, Müller, *Ber.*, 1904, **37**, 1339.

### Quinone.

See p-Benzoquinone.

### Quinone-anilide.

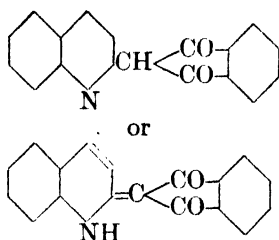
See 2:5-Dianilino-p-benzoquinone.

### Quinophan.

See 2-Phenylquinoline-4-carboxylic Acid.

**Quinophenol.**

See 8-Hydroxyquinoline.

**Quinophthalone** $C_{18}H_{11}O_2N$ 

MW, 273

Golden-yellow needles from EtOH. M.p.  $241^\circ$  ( $234^\circ$ ). Sol. hot AcOH, hot  $Me_2CO$ ,  $CHCl_3$ , warm  $C_6H_6$ , toluene. Prac. insol. EtOH,  $Et_2O$ . Sol. conc. HCl and conc.  $H_2SO_4$  with red col. Sublimes. Warm  $HNO_3$  (D 1:2)  $\rightarrow$  quinaldinic + phthalic acids.

*Phenylhydrazone*: red needles from  $CHCl_3$ . M.p.  $206^\circ$ .

*Anil*: red needles from EtOH. M.p.  $232^\circ$ . Sol.  $CHCl_3$ . Spar sol. EtOH.

*N-Me*: orange-yellow needles or brownish-red prisms. M.p.  $249.5^\circ$ .

Jacobsen, Reimer, *Ber.*, 1883, **16**, 1082.

Eibner, *Ber.*, 1904, **37**, 3606.

Eibner, Lange, *Ann.*, 1901, **315**, 336.

Eibner, Hofmann, *Ber.*, 1904, **37**, 3015, 3018.

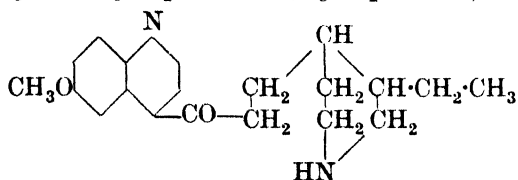
Kuhri, Bär, *Ann.*, 1935, **516**, 155.

Weno, Suzuki, *J. Soc. Chem. Ind. Japan*, 1933, **36**, Supplementary binding, 195.

**Quinosol.**

See under 8-Hydroxyquinoline.

**Quinotidine** (*Quinotoxine dihydride, quinicine dihydride, hydroquinotoxine, hydroquinicine*)

 $C_{20}H_{26}O_2N_2$ 

MW, 326

Yellow cryst. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , dil. acids.  $[\alpha]_D^{15} - 17^\circ$  in HCl. Gives yellow col. in  $H_2SO_4$  but no fluor.

$B_2H_2PtCl_6$ : orange cryst. +  $H_2O$ . Insol.  $H_2O$ .

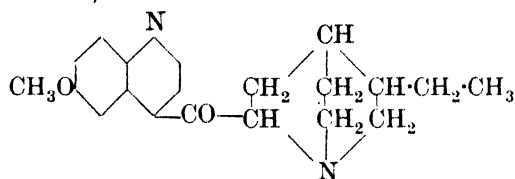
Hesse, *Ann.*, 1887, **241**, 273.

Rabe, Kindler, *Ber.*, 1919, **52**, 1844.

See also Kaufmann, Rothlin, Brunn-schweiler, *Ber.*, 1916, **49**, 2303.

**Quinotidine.**Hydroquinidine, *q.v.***Quinotine.**Hydroquinine, *q.v.*

**Quinotinone** (*Quininone dihydride, hydroquininone*)

 $C_{20}H_{24}O_2N_2$ 

MW, 324

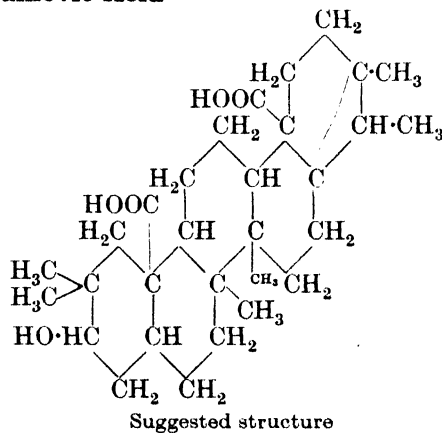
M.p.  $100^\circ$ .  $[\alpha]_D^{19} + 73.15^\circ$  in EtOH,  $[\alpha]_D^{20} + 65.0^\circ$  in 96% EtOH.

Rabe, Kindler, *Ber.*, 1919, **52**, 1845.

See also Kaufmann, Rothlin, Brunn-schweiler, *Ber.*, 1916, **49**, 2303.

**Quinotoxine.**

See Quinicine.

**Quinovic Acid** $C_{30}H_{46}O_5$ 

MW, 486

Needles. M.p.  $298^\circ$ . Spar. sol. hot EtOH, AcOH. Insol.  $H_2O$ . Sol.  $NH_3$  and alkalis.  $[\alpha]_D + 87-8^\circ$  in KOH. Decomp. at  $300^\circ \rightarrow$  pyroquinovic acid. Sol.  $H_2SO_4$  with evolution of CO.

*Di-Me ester*:  $C_{32}H_{50}O_5$ . MW, 514. M.p.  $173-4^\circ$ . *Acetyl*: m.p.  $208-9^\circ$ . *Benzoyl*: m.p.  $235-6^\circ$ .

*Di-Et ester*:  $C_{34}H_{54}O_5$ . MW, 542. Cryst. M.p.  $127-30^\circ$ . Sol. EtOH,  $Et_2O$ .

*Acetyl*: m.p.  $284^\circ$  decomp.

*Benzoyl*: m.p. 284° decomp.

Hlasiwetz, *Ann.*, 1851, **79**, 145.  
 Liebermann, Giesel, *Ber.*, 1883, **16**, 932.  
 Wieland, Erlenbach, *Ann.*, 1927, **453**, 83.  
 Wieland, Hoshino, *Ann.*, 1930, **479**, 179.  
 Wieland, Utzino, *Ann.*, 1931, **488**, 242.  
 Wieland, Kraus, *Ann.*, 1932, **497**, 140.  
 Wieland, Hartmann, Dietrich, *Ann.*, 1936, **522**, 191.

### Quinovin

$C_{36}H_{56}O_9$

MW, 632

$\alpha$ -.

Occurs with cinchona alkaloids as glucoside. Cryst. powder or needles from EtOH. Sol. EtOH. Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Prac. insol.  $H_2O$ .  $[\alpha]_D + 59.1^\circ$  ( $+ 56.6^\circ$ ) in EtOH. Bitter taste. Sol.  $NH_3$  and alkalis. Sol. conc.  $H_2SO_4$  with orange-yellow col. and evolution of CO. Reduces Fehling's. Hyd.  $\rightarrow$  quinovic acid + quinovose (isorhodoese).

$\beta$ -.

Occurs with alkaloids from the *Remijia* species. Plates from dil. EtOH. M.p. 235° decomp. Sol. EtOH. Insol.  $Et_2O$ , AcOEt.  $[\alpha]_D + 27.9^\circ$  in EtOH.

Hlasiwetz, *Ann.*, 1859, **111**, 182.

Rochleder, *J. prakt. Chem.*, 1867, **102**, 16.

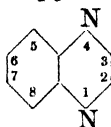
Liebermann, Giesel, *Ber.*, 1883, **16**, 928.

### Quinovose (*Chinovose*).

This sugar has been shown to be identical with isorhodoese, (*q.v.*).

See Votoček, Rác, *Chem. Abstracts*, 1929, **23**, 4449; 1932, **26**, 4307.

### Quinoxaline (*Benzpyrazine*, *phenpiazine*)



$C_8H_6N_2$

MW, 130

Cryst. M.p. 30.5° (28°). B.p. 225–6° (220–3°, 229.5°/760.3 mm.), 140°/40 mm.  $D_4^{25}$  1.1334.  $n_D^{25}$  1.6231. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ .  $KMnO_4 \rightarrow$  pyrazine-2 : 3-dicarboxylic acid.  $Na + EtOH \rightarrow$  1 : 2 : 3 : 4-tetrahydroquinoxaline.

$B.HCl$ : needles. Sinters at 170°. Decomp. at 184°. Sol.  $H_2O$ , EtOH.

$B.H_2SO_4$ : plates. M.p. 186–7°. Sol.  $H_2O$ . Mod. sol. EtOH.

$B_2.H_2PtCl_6$ : yellow needles.

*Oxalate*: needles. M.p. 169°. Spar. sol.  $H_2O$ .

*Methodide*: reddish-yellow plates from EtOH. M.p. 176° decomp. Sol.  $H_2O$ . Mod. sol. EtOH.

*Ethiodide*: red needles from EtOH. M.p. 146° decomp. Sol.  $H_2O$ .

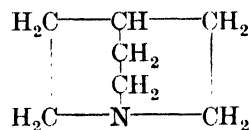
Hinsberg, *Ber.*, 1884, **17**, 320; *Ann.*, 1887, **237**, 334; 1896, **292**, 245.

Merz, Ris, *Ber.*, 1887, **20**, 1194.

### Quinoxyl.

*See* Yatren.

**Quinuclidine** (*Nuclidine*, 1 : 4-ethylenepiperidine)



$C_7H_{13}N$

MW, 111

Cryst. M.p. 158° (sealed tube). Very sol.  $H_2O$ , org. solvents. Very volatile. Ammoniacal odour. Does not decolourise  $KMnO_4$ .

$B.HAuCl_4$ : leaflets from  $H_2O$ . M.p. 271–3° decomp.

$B_2.H_2PtCl_6$ : leaflets from  $H_2O$ . M.p. 238–40° decomp.

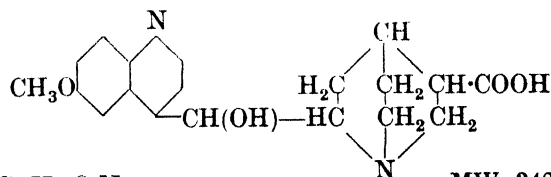
*Ethiodide*: plates from EtOH. M.p. 270–1°. Slightly hygroscopic.

*Ethochloroplatinate*: leaflets from hot  $H_2O$ . M.p. 271–2° (212°) decomp.

*Picrate*: pale yellow needles from EtOH. M.p. 275–6°. Sol. 35–40 parts hot EtOH.

Meisenheimer, Neresheimer, Schneider, *Ann.*, 1920, **420**, 213.

### Quitenine



$C_{19}H_{22}O_4N_2$

MW, 342

Prisms + 4 $H_2O$  from dil. EtOH. M.p. 286° decomp. (228° anhyd.). Loses  $H_2O$  at 110°. Sol. acids and alkalis. Spar. sol.  $H_2O$ . Insol.  $Et_2O$ . Sol. in dil.  $H_2SO_4$  shows blue fluor.  $[\alpha]_D - 143^\circ$  in EtOH. Heat with  $H_2SO_4 \rightarrow$  quitenicine. HI  $\rightarrow$  quitenol +  $CH_3I$ . Gives thalleioquin reaction.

$B_3.2H_2SO_4$ : prisms + 15 $H_2O$ . Sol.  $H_2O$ . Spar. sol. EtOH.

$B.H_2PtCl_6$ : yellow leaflets + 3 $H_2O$ . Spar. sol.  $H_2O$ .

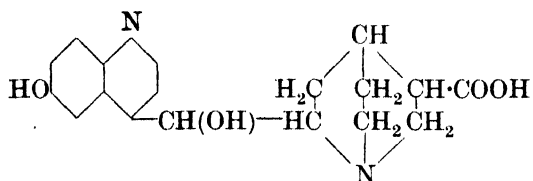
*Et ester*: needles. M.p. 198°. Insol.  $H_2O$ . *Ethiodide*: prisms. M.p. 210°.

Skraup, *Ann.*, 1879, **199**, 348.

Bucher, *Monatsh.*, 1893, **14**, 598.

Giemsa, Oesterlin, *Ber.*, 1931, **64**, 60.

Quitenol



$C_{18}H_{20}O_4N_2$

MW, 328

Needles +  $H_2O$ . Decomp. above  $270^\circ$  without melting. Sol. acids, alkalis. Pptd. from alk. sol. by  $CO_2$ . Prac. insol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ .  $FeCl_3$  in dil.  $HCl$  gives red col. Gives thalleioquin reaction.

$B, 2HCl$ : plates +  $H_2O$ .

$B, H_2PtCl_6$ : yellow prisms.

$B, H_2SO_4$ : yellow plates +  $H_2O$ .

*Me ether*: see Quitenine.

Bucher, *Monatsh.*, 1893, 14, 604.

R

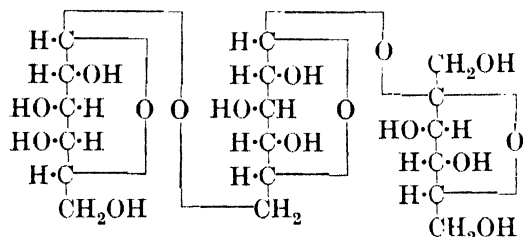
Racemic Acid.

See Tartaric Acid.

R-Acid.

See 2-Naphthol-3 : 6-disulphonic Acid.

Raffinose (*Melitriose, gossypose*)



$C_{18}H_{32}O_{16}$

MW, 504

Occurs in sugar beet, cotton seed, manna, etc. White needles or prisms +  $5H_2O$ . M.p.  $80^\circ$ , anhyd.  $118-19^\circ$ . Slight sweet taste.  $[\alpha]_D^{20} + 104^\circ$  in  $H_2O$  (hydrate),  $+ 123^\circ$  in  $H_2O$  (anhyd.).  $D^{20} 1.465$ . Sol.  $H_2O$ . Mod. sol.  $MeOH$ . Insol.  $EtOH$ ,  $Et_2O$ . Does not reduce Fehling's. Does not form an osazone. Hyd. by dil. acids  $\rightarrow$  galactose + glucose + fructose. Emulsin  $\rightarrow$  sucrose + galactose. Invertase or raffinase  $\rightarrow$  melibiose + fructose.  $HNO_3 \rightarrow$  saccharic and mucic acids.

*Undeca-acetyl*: cryst. from  $EtOH$ . M.p.  $99-101^\circ$ .  $[\alpha]_D^{20} + 92.2^\circ$ . Sol.  $EtOH$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $CS_2$ , ligroin.

*Octabenzoyl*: cryst. from  $AcOH$ . M.p.  $98^\circ$ .  $[\alpha]_D^{18.5} + 155.3^\circ$  in  $AcOH$ .

*Undecabenzoyl*: m.p.  $113^\circ$ .  $[\alpha]_D^{20.5} + 106.8^\circ$ . Sol.  $CHCl_3$ ,  $AcOEt$ . Mod. sol.  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ , hot  $MeOH$ , hot  $EtOH$ .

*Undeca-p-chlorobenzoyl*: m.p.  $130-2^\circ$ .  $[\alpha]_D^{18} + 96.8^\circ$ .

*Undeca-p-bromobenzoyl*: m.p.  $138^\circ$ .  $[\alpha]_D^{20.5} + 85.2^\circ$ .

*Undeca-Me ether*: b.p.  $238-40^\circ/0.02$  mm.

Harding, *Sugar*, 1923, 25, 308.

Englis, Decker, Adams, *J. Am. Chem. Soc.*, 1925, 47, 2724.

Hudson, Harding, *J. Am. Chem. Soc.*, 1914, 36, 2110.

Vogel, Pictet, *Helv. Chim. Acta*, 1928, 11, 898.

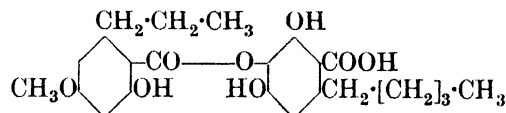
Haworth, Charlton, Hickinbottom, *J. Chem. Soc.*, 1927, 1527.

Hungerford, Nees, *Ind. Eng. Chem.*, 1934, 26, 462.

Ramalic Acid.

See Obtusatic Acid.

Ramalinolic Acid



$C_{23}H_{28}O_8$

MW, 432

Prisms from  $C_6H_6$ . M.p.  $164^\circ$ . Sol.  $EtOH$ ,  $Et_2O$ ,  $Me_2CO$ . Spar. sol.  $C_6H_6$ ,  $CHCl_3$ .  $FeCl_3 \rightarrow$  violet col. in  $EtOH$ .

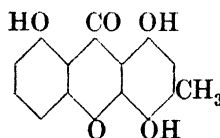
*Tri-Me ether Me ester*:  $C_{27}H_{36}O_8$ . MW, 488. M.p.  $75^\circ$ .

Asahina, Kusaka, *Ber.*, 1936, 69, 1896.

Ratanhin.

See Surinamine.

**Ravenelin** (1 : 4 : 5-Trihydroxy-2-methylxanthone)



$C_{14}H_{10}O_5$

MW, 258

Found in *Helminthosporium Ravenlii*, C., and *H. Turcicum*, P. Yellow prismatic needles from  $\text{Me}_2\text{CO}-\text{CHCl}_3$ . M.p. 267–8°. Sol. Py, hot AcOH. Mod. sol. hot EtOH,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{C}_6\text{H}_6$ , pet. ether. Hot  $\text{Na}_2\text{CO}_3$ . Aq.  $\rightarrow$  brownish-yellow sol. NaOH  $\rightarrow$  yellow sol.  $\rightarrow$  deep brown on shaking in air. Alc.  $\text{FeCl}_3$   $\rightarrow$  intense greenish-brown col. Conc.  $\text{H}_2\text{SO}_4$   $\rightarrow$  orange-yellow sol.  $\rightarrow$  orange-brown on heating.

*Di-Me ether*:  $\text{C}_{16}\text{H}_{14}\text{O}_5$ . MW, 286. Yellow needles from EtOH. M.p. 285–7°.

*Tri-Me ether*:  $\text{C}_{17}\text{H}_{16}\text{O}_5$ . MW, 300. Prisms from EtOH. M.p. 178–9°. Conc. HCl  $\rightarrow$  intense orange sol.  $\text{B.HCl, FeCl}_3$ : red prisms from AcOH. M.p. 174–5°.

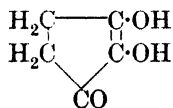
*Triacetyl*: needles from AcOH. M.p. 204–5°. Sol. hot EtOH,  $\text{C}_6\text{H}_6$ .

*Tribenzoyl*: prismatic needles from Py. M.p. 255°. Sol. warm Py. Mod. sol. hot AcOH. Spar. sol. boiling EtOH.

*Trianisoyl*: needles from AcOH. M.p. 216–18°.

Raistrick, Robinson, White, *Biochem. J.*, 1936, 30, 1303.

#### Reductic Acid (1 : 2-Dihydroxycyclopent- one-3)



$\text{C}_5\text{H}_6\text{O}_3$  MW, 114

Yellow cryst. from AcOEt. M.p. 213–213.5°. decomp. (207.5°). Sol.  $\text{H}_2\text{O}$ , EtOH, MeOH. Spar. sol.  $\text{Et}_2\text{O}$ , AcOEt,  $\text{Me}_2\text{CO}$ . Insol.  $\text{C}_6\text{H}_6$ .

*Acetyl deriv.*: m.p. 195°.

*Diacetyl deriv.*: oil. B.p. 112°/0.2 mm.

*Oxazone*: m.p. 246°.

*Me ether*:  $\text{C}_6\text{H}_8\text{O}_3$ . MW, 128. Needles. M.p. 138°.

*Di-Me ether*:  $\text{C}_7\text{H}_{10}\text{O}_3$ . MW, 142. B.p. 120°/12 mm.

Ilgami, *Chem. Abstracts*, 1934, 28, 7259.

Reichstein, Oppenauer, *Helv. Chim. Acta*, 1934, 17, 390.

#### Regularobufagin

$\text{C}_{25}\text{H}_{34}\text{O}_6$  MW, 430

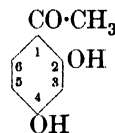
Prisms from EtOH.Aq. M.p. 235–6°.

*Diacetyl deriv.*: m.p. 224–5° (196–7°).

Kotake, *J. Chem. Soc. Japan*, 1934, 55, 179.

Jensen, *J. Am. Chem. Soc.*, 1935, 57, 1765.

#### Resacetophenone (4-Aceto-sorcinol, 2 : 4-di- hydroxyacetophenone)



$\text{C}_8\text{H}_8\text{O}_3$

MW, 152

Leaflets or needles. M.p. 147° (145–6°).  $\text{FeCl}_3$   $\rightarrow$  red col.

*2-Acetyl*: m.p. 119–20°.

*4-Acetyl*: m.p. 74°. *2-Benzoyl*: m.p. 67°.

*Diacetyl*: m.p. 38°.

*4-Benzoyl*: m.p. 106–7°. *2-Anisoyl*: m.p. 109–10°.

*Dibenzoyl*: m.p. 80–1°.

*Dianisoyl*: m.p. 118°.

*Diveratroyl*: m.p. 151–2°.

*Dicinnamoyl*: m.p. 131°.

*Oxime*: m.p. 198–200° decomp. *Acetyl*: m.p. 174–5° decomp.

*Semicarbazone*: m.p. 214–20° decomp.

*Phenylhydrazone*: m.p. 159°.

*2-Me ether*: see Isopaeonol (Isopeonol).

*4-Me ether*: see Peonol.

*Di-Me ether*: 2 : 4-dimethoxyacetophenone, peonol methyl ether, isopeonol methyl ether.  $\text{C}_{10}\text{H}_{12}\text{O}_3$ . MW, 180. M.p. 44° (40°). *Oxime*: m.p. 125°.

*2-Me*: 4-Et ether: isopeonol ethyl ether.  $\text{C}_{11}\text{H}_{14}\text{O}_3$ . MW, 194. Needles from EtOH. M.p. 49°.

*4-Me*: 2-Et ether: peonol ethyl ether. Needles from EtOH. M.p. 70°.  $D^{20}_D$  1.0571.  $n^{20}_D$  1.51434.

*4-Et ether*:  $\text{C}_{10}\text{H}_{12}\text{O}_3$ . MW, 180. Needles from EtOH. M.p. 49°.

*Di-Et ether*: 2 : 4-diethoxyacetophenone.  $\text{C}_{12}\text{H}_{16}\text{O}_3$ . MW, 208. M.p. 74°. *Oxime*: m.p. 122°.

*4-Propyl ether*:  $\text{C}_{11}\text{H}_{14}\text{O}_3$ . MW, 194. M.p. 25°.

*Dipropyl ether*:  $\text{C}_{14}\text{H}_{20}\text{O}_3$ . MW, 236. M.p. 26°.

*4-Butyl ether*:  $\text{C}_{12}\text{H}_{16}\text{O}_3$ . MW, 208. M.p. 43°.

*Dibutyl ether*:  $\text{C}_{16}\text{H}_{24}\text{O}_3$ . MW, 264. M.p. 32°.

*4-α-Glycerol ether*:  $\text{C}_{11}\text{H}_{14}\text{O}_5$ . MW, 226. M.p. 88°. *Phenylhydrazone*: m.p. 119–20°.

Robinson, Shah, *J. Chem. Soc.*, 1934, 1494.

Gulati, Venkataraman, *J. Chem. Soc.*, 1931, 2376.

Kostanecki, Tambor, *Ber.*, 1895, **28**, 2306.

Schaffer, U.S.P., 1,745,507, (*Chem. Abstracts*, 1930, **24**, 1707).

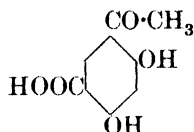
Kondo, Nakagawa, *Chem. Abstracts*, 1931, **25**, 515.

Cox, *Rec. trav. chim.*, 1931, **50**, 850.

Baker, *J. Chem. Soc.*, 1934, 1691.

Nadkarni, Wheeler, *J. Chem. Soc.*, 1936, 589.

**Resacetophenone-5-carboxylic Acid** (2:4-Dihydroxyacetophenone-5-carboxylic acid, 2:4-dihydroxy-5-acetobenzoic acid)



$C_9H_8O_5$

MW, 196

Needles from  $Me_2CO$ . M.p.  $256^\circ$  decomp.

*Me ester*:  $C_{10}H_{10}O_5$ . MW, 210. Needles from  $C_6H_6$ . M.p.  $124-5^\circ$ . *Hydrazone*: (i) m.p.  $138^\circ$ . (ii) M.p.  $170^\circ$ .

*Di-Me ether*:  $C_{11}H_{12}O_5$ . MW, 224. Prisms from  $MeOH$ . M.p.  $231-3^\circ$ .

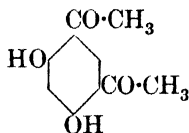
*Et ester*:  $C_{11}H_{12}O_5$ . MW, 224. M.p.  $94^\circ$ .

*Di-Et ether*:  $C_{15}H_{20}O_5$ . MW, 280. Needles from ligroin. M.p.  $96-7^\circ$ .

*p-Bromophenylhydrazone*: m.p.  $243^\circ$ .

Liebermann, Lindenbaum, *Ber.*, 1908, **41**, 1610.

**Resodiacetophenone** (4:6-Diacetoresorcinol)



$C_{10}H_{10}O_4$

MW, 194

Needles from  $EtOH$ . M.p.  $182^\circ$ . Sol. hot  $EtOH$ , hot  $C_6H_6$ , warm  $Et_2O$ , warm  $AcOH$ .

*Diacetyl*: m.p.  $120^\circ$ .

*Dibenzoyl*: m.p.  $118^\circ$ .

*Dioxime*: m.p.  $242^\circ$ .

*Phenylhydrazone*: m.p.  $233^\circ$ . *Monoacetyl deriv.*: m.p.  $191-2^\circ$ . *Monobenzoyl deriv.*: m.p.  $214-15^\circ$ .

*Diphenylhydrazone*: m.p.  $291^\circ$ .

*Mono-Me ether*:  $C_{11}H_{12}O_4$ . MW, 208. M.p.  $121-5^\circ$ . *Diphenylhydrazone*: m.p.  $245-6^\circ$ , decomp.

*Di-Me ether*:  $C_{12}H_{14}O_4$ . MW, 222. M.p.  $171-5^\circ$ .

*Mono-Et ether*:  $C_{12}H_{14}O_4$ . MW, 222. M.p.  $109^\circ$ .

*Di-Et ether*:  $C_{14}H_{18}O_4$ . MW, 250. M.p.  $156^\circ$ .

*Me-Et ether*:  $C_{13}H_{16}O_4$ . MW, 236. M.p.  $152^\circ$ .

*Monopropyl ether*:  $C_{13}H_{16}O_4$ . MW, 236. M.p.  $94-5^\circ$ .

*Me-Propyl ether*:  $C_{14}H_{18}O_4$ . MW, 250. M.p.  $125-5^\circ$ .

*Et-Propyl ether*:  $C_{15}H_{20}O_4$ . MW, 264. M.p.  $95^\circ$ .

*Dipropyl ether*:  $C_{16}H_{22}O_4$ . MW, 278. M.p.  $86^\circ$ .

*Monoisopropyl ether*: m.p.  $97^\circ$ .

*Et-Isopropyl ether*: m.p.  $101-5^\circ$ .

*Propyl-Isopropyl ether*: m.p.  $78^\circ$ .

*Di-isopropyl ether*: m.p.  $126-5^\circ$ .

*Monobutyl ether*:  $C_{14}H_{18}O_4$ . MW, 250. M.p.  $63^\circ$ .

*Me-Butyl ether*:  $C_{15}H_{20}O_4$ . MW, 264. M.p.  $117^\circ$ .

*Et-Butyl ether*:  $C_{16}H_{22}O_4$ . MW, 278. M.p.  $104^\circ$ .

*Propyl-Butyl ether*:  $C_{17}H_{24}O_4$ . MW, 292. M.p.  $61-5^\circ$ .

*Isopropyl-Butyl ether*: m.p.  $76^\circ$ .

*Dibutyl ether*:  $C_{18}H_{26}O_4$ . MW, 306. M.p.  $92-5^\circ$ .

*Mono-isobutyl ether*: m.p.  $88^\circ$ .

*Me-Isobutyl ether*: m.p.  $102-5^\circ$ .

*Et-Isobutyl ether*: m.p.  $72^\circ$ .

*Propyl-Isobutyl ether*: m.p.  $64^\circ$ .

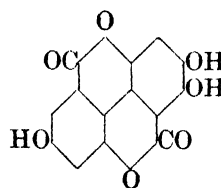
*Isopropyl-Isobutyl ether*: m.p.  $80^\circ$ .

*Butyl-Isobutyl ether*: m.p.  $78^\circ$ .

*Di-isobutyl ether*: m.p.  $99^\circ$ .

Baker, *J. Chem. Soc.*, 1934, 71 (*Bibl.*); *ibid.*, 1684.

**Resoflavin** (3:4:6:4':6'-Pentahydroxydiphenic acid dilactone)



$C_{14}H_6O_7$

MW, 286

Yellow cryst. from  $EtOH$ . Decomp. about  $380^\circ$ . Zn dist.  $\rightarrow$  fluorene.

*Triacetyl*: m.p.  $275-9^\circ$ .

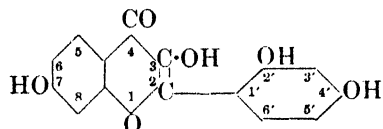
*Tri-Me ether*:  $C_{17}H_{12}O_7$ . MW, 328. M.p.  $286-8^\circ$ .

Badische, D.R.P., 85,390.

Herzig, Tscherne, *Ann.*, 1907, 351, 30.



**Resomorin** (3 : 7 : 2' : 4'-Tetrahydroxyflavone, 7 : 2' : 4'-trihydroxyflavonol)



$C_{15}H_{10}O_6$

MW, 286

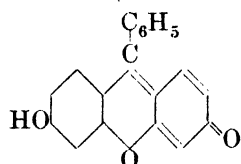
Cryst. Very sol. EtOH.

7 : 2' : 4'-Tri-Me ether :  $C_{18}H_{16}O_6$ . MW, 328. Cryst. from  $C_6H_6$  or EtOH. M.p. 205°. Conc.  $H_2SO_4 \rightarrow$  yellowish sol. with intense bluish-green fluor. Acetyl : prisms from EtOH.Aq. M.p. 189-91°.

Tetra-acetyl : needles from EtOH.Aq. M.p. 129-30°.

v. Kostanecki, Lampe, Triulzi, *Ber.*, 1906, 39, 94.

**Resorcin-benzein** (*Resorcinol-benzein*)



$C_{19}H_{12}O_3$

MW, 288

Red needles from  $PhNO_2$ . M.p. 330-1°. Sol. EtOH,  $Me_2CO$ ,  $PhNO_2$ , aniline, Py. Spar. sol.  $Et_2O$ ,  $CHCl_3$ . Sols. fluoresce.

6-Acetyl : m.p. 197°.

6-Me ether :  $C_{20}H_{14}O_3$ . MW, 302. Red needles from  $Et_2O$ . M.p. 206° (204°).

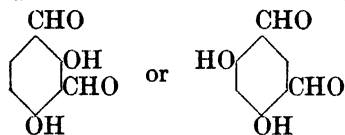
Liebig, *J. prakt. Chem.*, 1912, 85, 244.

Gomberg, West, *J. Am. Chem. Soc.*, 1912, 34, 1568.

Pope, Howard, *J. Chem. Soc.*, 1910, 97, 1026.

Moir, *Chem. Abstracts*, 1923, 17, 2283.

**Resorcindialdehyde** (2 : 4 (or 4 : 6)-Dihydroxyisophthalaldehyde, resorcendialdehyde)



$C_8H_6O_4$

MW, 166

Needles from hot  $H_2O$ . M.p. 127°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Sublimes.

Dioxime : m.p. 209°.

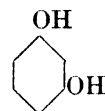
Me ether :  $C_9H_8O_4$ . MW, 180. (i) Needles from hot  $H_2O$ . M.p. 179°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , AcOH. Insol. ligroin. (ii)

Needles from hot  $H_2O$ . M.p. 88-9°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin. Spar. sol.  $H_2O$ . Both forms volatile in steam.

Tiemann, Lewy, *Ber.*, 1877, 10, 2211.

Tiemann, Parrisius, *Ber.*, 1880, 13, 2369.

**Resorcinol** (1 : 3-Dihydroxybenzene)



$C_6H_6O_2$

MW, 110

Needles from  $C_6H_6$ . Plates from  $H_2O$ . (i) M.p. 110°, stable. (ii) M.p. 108-108.5°, labile. B.p. 273° (276.5°/759.7 mm.), 178°/16 mm. Sol.  $H_2O$ , EtOH,  $Et_2O$ . Mod. sol.  $CHCl_3$ ,  $CS_2$ . Spar. sol.  $C_6H_6$ .  $FeCl_3 \rightarrow$  violet col. Heat of comb.  $C_p$  683.1 Cal.  $k = 3.6 \times 10^{-10}$  at 18°. Reduces Tollen's and Fehling's. Sweet taste.

Mono-acetyl : curesol. B.p. 283°.

Diacetyl : b.p. 278°, 273°/708 mm.

Dichloroacetyl : m.p. 71.5-72° (76°). B.p. 191-2°/15 mm.

Di-iodoacetyl : m.p. 59-61°.

Di-1-bromopropionyl : m.p. 66°. B.p. 217-20°/10 mm.

Di-1-bromobutyryl : b.p. 225-7°/19 mm.

Di-1-bromoisobutyryl : b.p. 227-8°/20 mm.

Di-1-bromoisovaleryl : b.p. 222-8°/15 mm.

Penta-acetyl- $\beta$ -glucoside : m.p. 118-19°.  $[\alpha]_D^{25} - 40.1^\circ$  in  $C_6H_6$ .

Monobenzoyl : m.p. 135-6° (133°).

Dibenzoyl : m.p. 117°.

Mono-salicyloyl : m.p. 141° (139°).

Disalicyloyl : m.p. 111°.

Di-p-nitrobenzoyl : m.p. 182°.

Dicinnamoyl : m.p. 119.5-120°.

Monohippuryl : see Hippurylresorcinol.

Di-benzenesulphonyl : m.p. 69-70°.

Di-p-toluenesulphonyl : m.p. 80-1°.

Di-d-camphor- $\beta$ -sulphonyl : m.p. 129-30°.

Picrate : m.p. 89-90°.

Mono-Me ether : m-hydroxyanisole, m-methoxyphenol.  $C_7H_8O_2$ . MW, 124. B.p. 244°, 144°/25 mm., 102°/5 mm. Acetyl : b.p. 254-6°.

Di-Me ether : 1 : 3-dimethoxybenzene.  $C_8H_{10}O_2$ . MW, 138. M.p. -52°. B.p. 216.5-217.7°.  $D_4^{25}$  1.0705.

Mono-Et ether : m-hydroxyphenetole, m-ethoxyphenol.  $C_8H_{10}O_2$ . MW, 138. B.p. 246-7°, 117°/5.5 mm.

Di-Et ether : 1 : 3-diethoxybenzene.  $C_{10}H_{14}O_2$ . MW, 166. M.p. 12.4°. B.p. 234.4-235.2°/756 mm.

Me-Et ether :  $C_9H_{12}O_2$ . MW, 152. B.p. 216°.

*Mono-propyl ether*:  $C_9H_{12}O_2$ . MW, 152. B.p.  $120^\circ/5$  mm.

*Me-Propyl ether*:  $C_{10}H_{14}O_2$ . MW, 166. B.p.  $226^\circ$ .

*Dipropyl ether*:  $C_{12}H_{18}O_2$ . MW, 194. B.p.  $251^\circ$ .

*Monobutyl ether*:  $C_{10}H_{14}O_2$ . MW, 166. B.p.  $130^\circ/5$  mm.

*Me-Isobutyl ether*:  $C_{11}H_{16}O_2$ . MW, 180. B.p.  $234^\circ$ .

*Monoamyl ether*:  $C_{11}H_{16}O_2$ . MW, 180. B.p.  $140^\circ/5$  mm.

*Mono-isoamyl ether*: b.p.  $138^\circ/5$  mm.

*Di-isoamyl ether*:  $C_{16}H_{26}O_2$ . MW, 250. M.p.  $47^\circ$ .

*Monoheptyl ether*:  $C_{12}H_{18}O_2$ . MW, 194. B.p.  $145^\circ/5$  mm.

*Monoheptyl ether*:  $C_{13}H_{20}O_2$ . MW, 208. B.p.  $160^\circ/5$  mm.

*Mono-octyl ether*:  $C_{14}H_{22}O_2$ . MW, 222. B.p.  $170^\circ/5$  mm.

*Mono-nonyl ether*:  $C_{15}H_{24}O_2$ . MW, 236. B.p.  $171^\circ/4.5$  mm.

*Di-trichlorovinyl ether*:  $C_{10}H_4O_2Cl_6$ . MW, 369. M.p.  $53-4^\circ$ .

*Phenyl ether*: see 3-Hydroxydiphenyl Ether.

*Diphenyl ether*:  $C_{18}H_{14}O_2$ . MW, 262. M.p.  $61.5^\circ$ .

*Di-2:4-dinitrophenyl ether*:  $C_{18}H_{10}O_{10}N_4$ . MW, 442. M.p.  $184^\circ$ .

*Monobenzyl ether*:  $C_{13}H_{12}O_2$ . MW, 200. M.p.  $69.2^\circ$ . B.p.  $200^\circ/5$  mm.

*Mono-p-chlorobenzyl ether*:  $C_{13}H_{11}O_2Cl$ . MW, 234.5. M.p.  $76^\circ$ . B.p.  $235^\circ/13$  mm.

*Dibenzyl ether*:  $C_{20}H_{18}O_2$ . MW, 290. M.p.  $76^\circ$ .

*Mono-phenylethyl ether*:  $C_{14}H_{14}O_2$ . MW, 214. M.p.  $44^\circ$ . B.p.  $202^\circ/6$  mm.

*Mono-phenylpropyl ether*:  $C_{15}H_{16}O_2$ . MW, 228. B.p.  $220^\circ/5.5$  mm.

*Mono-cyclohexyl ether*:  $C_{12}H_{16}O_2$ . MW, 192. B.p.  $160^\circ/6$  mm.

*Me- $\alpha$ -phenylvinyl ether*:  $C_{15}H_{14}O_2$ . MW, 226. B.p.  $199-200^\circ/16$  mm.

Knoll, D.R.P., 281,099, (*Chem. Zentr.*, 1915, I, 179).

Phillips, Gibbs, *Ind. Eng. Chem.*, 1920, 12, 857.

Carr, Dahlen, U.S.P., 1,999,955, (*Chem. Abstracts*, 1935, 29, 4029).

Gallay, U.S.P., 1,956,570, (*Chem. Abstracts*, 1934, 28, 4073).

Hayashi, *Chem. Abstracts*, 1934, 28, 1679.

Ruth, *Chem. Abstracts*, 1933, 27, 5316.

Klarman, Gatyas, Shternov, *J. Am. Chem. Soc.*, 1931, 35, 3397.

Fabre, *Ann. chim.*, 1922, 18, 49.

Dey, *J. Indian Chem. Soc.*, 1935, 12, 685.

Dict. of Org. Comp.—III.

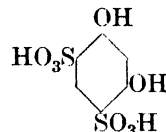
### Resorcinol-carboxylic Acid.

See Resorcylic Acid.

### Resorcinol-dicarboxylic Acid.

See 4:6-Dihydroxyisophthalic Acid and 2:6-Dihydroxyterephthalic Acid.

### Resorcinol-4:6-disulphonic Acid



$C_6H_6O_8S_2$  MW, 270

Needles +  $2H_2O$ . Decomp. at  $100^\circ$ . Sol.  $H_2O$ , EtOH. Insol. Et<sub>2</sub>O.

*Dichloride*:  $C_6H_4O_6Cl_2S_2$ . MW, 307. M.p.  $178-9^\circ$ . *Di-Me ether*:  $C_8H_8O_6Cl_2S_2$ . MW, 335.

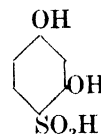
Yellow prisms from AcOEt. M.p.  $175-8^\circ$ . Sol. Et<sub>2</sub>O,  $CHCl_3$ , AcOH,  $CS_2$ .

*Diamide*:  $C_6H_8O_6N_2S_2$ . MW, 268. Does not melt below  $300^\circ$ .

*Dianilide*: m.p.  $262^\circ$ .

Blumenstock, *Monatsh.*, 1925, 46, 499.

### Resorcinol-4-sulphonic Acid



$C_6H_6O_5S$  MW, 190

*K salt*: prisms +  $2H_2O$  from  $H_2O$ .

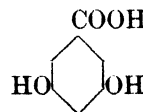
*Ba salt*: hygroscopic cryst.

Darzens, Dubois, *Bull. soc. chim.*, 1892, 7, 713.

### $\beta$ -Resorcylicyclohexane.

See 4-Hexahydrobenzoylresorcinol.

$\alpha$ -Resorcylic Acid (3:5-Dihydroxybenzoic acid, resorcinol-5-carboxylic acid)



$C_7H_6O_4$  MW, 154

Cryst. +  $1\frac{1}{2}H_2O$  from  $H_2O$ . M.p.  $232-3^\circ$  ( $237-40^\circ$ ) anhyd. Sol. EtOH, Et<sub>2</sub>O, hot  $H_2O$ .  $k = 9.1 \times 10^{-5}$  at  $25^\circ$ . Oleum or hot conc.  $H_2SO_4 \rightarrow 1:2:4:5:6:8$ -hexahydroxy-anthraquinone.

*Diacetyl*: m.p.  $156-7^\circ$ .

*Dicarbomethoxyl*: m.p.  $161-4^\circ$ .

*Me ester*:  $C_8H_8O_4$ . MW, 168. Leaflets. M.p.  $163-5^\circ$ . *Mono-Me ether*:  $C_9H_{10}O_4$ . MW, 182. B.p.  $375^\circ$  decomp. *Di-Me ether*:

$C_{10}H_{12}O_4$ . MW, 196. M.p. 42–4°. B.p. 298°, 157°/125 mm.

*Di-Me ether*: see 3:5-Dimethoxybenzoic Acid.

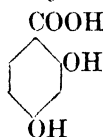
*Di-Et ether*:  $C_{11}H_{14}O_4$ . MW, 210. M.p. 87–8°. *Et ester*:  $C_{13}H_{18}O_4$ . MW, 238. M.p. 19–20°. B.p. 212°/50 mm.

Zollinger, Roehling, U.S.P., 1,321,271, (Chem. Abstracts, 1920, 14, 186).

Boehringer, D.R.P., 286,266, (Chem. Zentr., 1915, II, 566).

Barth, Senhofer, Ann., 1871, 159, 222.

**$\beta$ -Resorcylic Acid** (2:4-Dihydroxybenzoic acid, resorcinol-4-carboxylic acid)



$C_7H_6O_4$  MW, 154

Cryst. from  $H_2O$ . M.p. 213° (rapid heat.), (204–6°, 197° decomp.). Sol. EtOH, Et<sub>2</sub>O, hot  $H_2O$ .  $k = 5.16 \times 10^{-4}$  at 25°. Heat of comb.  $C_p$  676.9 Cal.,  $C_v$  677.2 Cal. Heat  $\rightarrow$  resorcinol.  $FeCl_3 \rightarrow$  red col.  $NaOCl \rightarrow$  violet col.

2-Acetyl: m.p. 167–8° decomp. (rapid heat.).

4-Acetyl: m.p. 152–3°.

Diacetyl: m.p. 136–8°.

2-Acetyl-4-benzoyl: m.p. 148–9°.

*Di-carbomethoxyl*: m.p. 159°. *Chloride*: m.p. 86–7°.

2-Benzoyl: m.p. 160–1°. *Carbomethoxyl*: m.p. 148–9°.

4-Benzoyl: m.p. 193–4°.

*Me ester*:  $C_8H_8O_4$ . MW, 168. M.p. 118–19°.

*p-Nitrobenzyl ester*:  $C_{14}H_{11}O_6N$ . MW, 289. M.p. 189°.

2-Me ether:  $C_8H_8O_4$ . MW, 168. Leaflets from  $H_2O$ . Decomp. at 187–9° (rapid heat.).

4-Me ether: 2-hydroxyanisic acid, 4-methoxysalicylic acid. M.p. 157° (about 161°).

*Acetyl*: m.p. 145–7°. *Me ester*:  $C_9H_{10}O_4$ . MW, 182. Primula camphor. Occurs in root of *Primula veris*.<sup>\*</sup> Plates from ligroin. M.p. 49°.

*Et ester*:  $C_{10}H_{12}O_4$ . MW, 196. B.p. 272–4°.

*Di-Me ether*: see 2:4-Dimethoxybenzoic Acid.

2-Et ether:  $C_9H_{10}O_4$ . MW, 182. Needles from  $H_2O$ . M.p. 154°. Sol. EtOH, Et<sub>2</sub>O,  $C_6H_6$ , hot  $H_2O$ .

4-Et ether: *Me ester*:  $C_{10}H_{12}O_4$ . MW, 196. Plates from pet. ether. M.p. 77–9°. *Et ester*:  $C_{11}H_{14}O_4$ . MW, 210. Needles from EtOH. M.p. 53–4° (45°).

*Di-Et ether*:  $C_{11}H_{14}O_4$ . MW, 210. Needles from EtOH.Aq. M.p. 99°. *Me ester*:  $C_{12}H_{16}O_4$ . MW, 224. Cubes from EtOH.Aq. M.p. 51–4°.

*Chloride*:  $C_7H_5O_3Cl$ . MW, 172.5. F.p. –20°. B.p. 170°/12 mm.

*Amide*:  $C_7H_7O_3N$ . MW, 153. M.p. 221–2°.

*Nitrile*: 4-cyanoresorcinol.  $C_7H_5O_2N$ . MW, 135. M.p. 175°. Sol.  $H_2O$ , EtOH. *Diacetyl*: m.p. 72°.

*Anilide*: m.p. 126–7°.

Shoosmith, Haldane, J. Chem. Soc., 1924, 125, 113.

Miksic, J. prakt. Chem., 1928, 119, 218.

Clibbens, Nierenstein, J. Chem. Soc., 1915, 107, 1493; Organic Syntheses, 1930, X, 94.

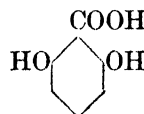
Robinson, Shah, J. Chem. Soc., 1934, 1496.

Mauthner, J. prakt. Chem., 1930, 124, 319.

Bergmann, Dangschat, Ber., 1919, 52, 371.

\*Note.—*Primula veris* is the synonym of three different *Primula* species. Since the authority is not given in the original paper it is impossible to assign the correct Index Kewensis name to this plant.

**$\gamma$ -Resorcylic Acid** (2:6-Dihydroxybenzoic acid, resorcinol-2-carboxylic acid)



$C_7H_6O_4$  MW, 154

Cryst. +  $1H_2O$  from  $H_2O$ . M.p. varies from 150 to 170° according to rate of heating  $\rightarrow$  resorcinol. Sol. EtOH, Et<sub>2</sub>O, hot  $H_2O$ .  $k = 5.0 \times 10^{-2}$  at 25°. Reduces warm Fehling's but not Tollen's.  $FeCl_3 \rightarrow$  violet to blue col.

*Me ester*:  $C_8H_8O_4$ . MW, 168. M.p. 67–8°.

*Me ether*:  $C_8H_8O_4$ . MW, 168. Cryst. from  $CHCl_3$ - $C_6H_6$ . M.p. 135°.

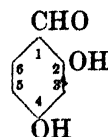
*Di-Me ether*: see 2:6-Dimethoxybenzoic Acid.

*Di-Et ether*:  $C_{11}H_{13}O_2N$ . MW, 191. M.p. 122°.

Brunner, Ann., 1907, 351, 320.

Mauthner, J. prakt. Chem., 1930, 124, 319.

**$\beta$ -Resorcylic Aldehyde** (2:4-Dihydroxybenzaldehyde)



$C_7H_6O_3$

MW, 138

Needles from  $\text{Et}_2\text{O}$ -ligroin. M.p. 135–6°. B.p. 220–8°/22 mm. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{AcOH}$ . Spar. sol.  $\text{C}_6\text{H}_6$ . Gives deep brown col. with  $\text{FeCl}_3$ , yellow ppt. with Schiff's reagent.

*Diacetyl*: m.p. 69°.

*Di-carbomethoxyl*: m.p. 72°. *Phenylhydrazone*: m.p. 138°. *Semicarbazone*: m.p. 185°.

*Oxime*: m.p. 192°.

*Semicarbazone*: decomp. at 260°.

*Phenylhydrazone*: m.p. 156–60° decomp.

unsym.-*Me-p-nitrophenylhydrazone*: m.p. 265°.

3 : 4-*Dichloro-6-nitrophenylhydrazone*: m.p. 251°.

*Anil*: m.p. 125–6°.

2-*Me ether*:  $\text{C}_8\text{H}_8\text{O}_3$ . MW, 152. M.p. 153°.

4-*Acetyl*: m.p. 86°.

4-*Me ether*: 2-hydroxyanisaldehyde, 4-methoxysalicylaldehyde. M.p. 40–2°.

*Di-Me ether*: 2-methoxyanisaldehyde.  $\text{C}_9\text{H}_{10}\text{O}_3$ . MW, 166. M.p. 68–9° (71°). B.p. 165°/10 mm.

4-*Et ether*: 4-ethoxysalicylaldehyde.  $\text{C}_9\text{H}_{10}\text{O}_3$ . MW, 166. M.p. 35°.

*Di-Et ether*:  $\text{C}_{11}\text{H}_{14}\text{O}_3$ . MW, 194. M.p. 71–2°.

Karrer, *Helv. Chim. Acta*, 1919, **2**, 89.

Weil, Traun, Marcel, *Ber.*, 1922, **55**, 2665.

Mitter, Suha, *J. Indian Chem. Soc.*, 1934, **11**, 257.

Hinkel, Ayling, Morgan, *J. Chem. Soc.*, 1932, 2798.

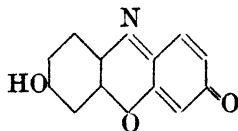
Shoosmith, Haldane, *J. Chem. Soc.*, 1923, **123**, 2704.

**$\gamma$ -Resorcylic Aldehyde** (2 : 6-Dihydroxybenzaldehyde).

M.p. 155–6°.

Shah, Laiwalla, *Chem. Zentr.*, 1937, **I**, 1679.

**Resorufine** (Azoresorufin, 9-hydroxy-3-isophenoxazone, diazoresorufine)



$\text{C}_{12}\text{H}_7\text{O}_3\text{N}$

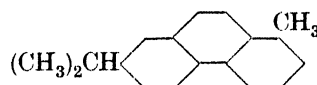
MW, 213

*Acetyl deriv.*: m.p. 221°.

*Et ether*:  $\text{C}_{14}\text{H}_{11}\text{O}_3\text{N}$ . MW, 241. Orange-red needles from  $\text{EtOH}$ . M.p. 225°.

Eichler, *J. prakt. Chem.*, 1934, **139**, 113.

**Retene** (1-Methyl-7-isopropylphenanthrene)



$\text{C}_{18}\text{H}_{18}$

MW, 234

Plates from  $\text{EtOH}$ . M.p. 100.5–101° (98–9°). B.p. 390°, 158–65°/0.2 mm. Sol.  $\text{C}_6\text{H}_6$ , ligroin,  $\text{CS}_2$ , hot  $\text{EtOH}$ , hot  $\text{AcOH}$ . Heat of comb.  $\text{C}_p$  2326.1 Cal.,  $\text{C}_v$  2323.6 Cal.

*Picrate*: m.p. 124–5°.

*Styphnate*: m.p. 141–2°.

$\text{C}_{18}\text{H}_{18}, \text{C}_6\text{H}_3(\text{NO}_3)_3$  1 : 3 : 5 : m.p. 139–40°.

Cheung, *Chem. Abstracts*, 1929, **23**, 4464, (Bibl.).

Haworth, Letsky, Mavin, *J. Chem. Soc.*, 1932, 1791.

Nagel, Körnchen, *Chem. Abstracts*, 1932, **26**, 1808.

Bardhan, Sengupta, *J. Chem. Soc.*, 1932, 2798.

Henke, Etzel, U.S.P., 1,881,565, (*Chem. Abstracts*, 1933, **27**, 516).

Nyman, *Chem. Zentr.*, 1936, **I**, 2348.

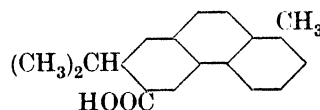
Ruzicka, Waldman, *Helv. Chim. Acta*, 1933, **16**, 847.

Hosking, McFadyen, *J. Soc. Chem. Ind.*, 1934, **53**, 195T.

Darzens, Lévy, *Chem. Zentr.*, 1937, **I**, 592.

Keimatsu, Ishiguro, *Chem. Abstracts*, 1935, **29**, 7323, (Refs.).

**Retene-6-carboxylic Acid** (1-Methyl-7-isopropylphenanthrene-6-carboxylic acid)



$\text{C}_{19}\text{H}_{18}\text{O}_2$

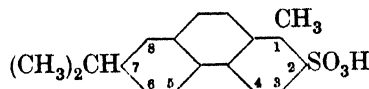
MW, 278

Needles from  $\text{C}_6\text{H}_6$ . M.p. 241–3° (238–238.5°).

Nyman, *Chem. Zentr.*, 1936, **I**, 2349.

Adelson, Bogert, *J. Am. Chem. Soc.*, 1936, **58**, 654.

**Retene-2(?) -sulphonic Acid**



$\text{C}_{18}\text{H}_{18}\text{O}_3\text{S}$

MW, 314

M.p. 188–9°.

*Me ester*:  $\text{C}_{19}\text{H}_{20}\text{O}_3\text{S}$ . MW, 328. M.p. 164–6°.

*Et ester*:  $C_{20}H_{20}O_3S$ . MW, 342. M.p. 137·5–138·5°.

*Chloride*:  $C_{18}H_{17}O_2ClS$ . MW, 332·5. Yellow scales from  $Et_2O$ . M.p. 135–6°.

Komppa, Fogelberg, *J. Am. Chem. Soc.*, 1932, **54**, 2907.

### Retene-6-sulphonic Acid.

M.p. 121–3°.

*Me ester*: m.p. 117–19°.

*Et ester*: m.p. 114–15°.

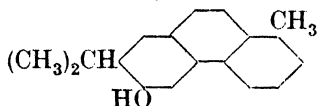
*Chloride*: yellow needles from  $C_6H_6$ . M.p. 146–147·5°.

*Amide*:  $C_{18}H_{19}O_2NS$ . MW, 313. M.p. 206–207·5°.

See previous reference and also

Hasselstrom, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 1580.

### 6-Retenol (6-Hydroxyretene)



$C_{18}H_{18}O$  MW, 250

Yellow scales from xylene. M.p. 163·5–164·5°.

*Acetyl*: m.p. 134–5°.

Adelson, Bogert, *J. Am. Chem. Soc.*, 1936, **58**, 653.

### Retronecine (Senecifolinene?)

$C_8H_{13}O_2N$  MW, 155

M.p. 121–2°. Sol.  $H_2O$ ,  $EtOH$ . Mod. sol.  $Me_2CO$ ,  $CHCl_3$ . Spar. sol.  $Et_2O$ .  $[\alpha]_D^{20} + 50-2^\circ$  in  $EtOH$ . Alkaline. Forms no picrate.

$B, HCl$ : m.p. 162–3° (161°).  $[\alpha]_D^{15} - 16^\circ$  in  $EtOH$ .

*Aurichloride*: m.p. 146°.

*Diacetyl deriv.*: picrate, m.p. 146°. *Methiodide*: m.p. 118–20°.

Barger, Seshadri, Watt, Yabuta, *J. Chem. Soc.*, 1935, 13.

Barger, Blackie, *J. Chem. Soc.*, 1936, 744.

### Retrorsine

$C_{18}H_{25}O_6N$  MW, 351

Occurs in *Senecio barbellatus*, D.C. Leaflets from  $AcOEt$ . M.p. 214–15° (212°). Sol.  $EtOH$ ,  $CHCl_3$ . Spar. sol.  $H_2O$ ,  $Et_2O$ ,  $AcOEt$ ,  $Me_2CO$ .  $[\alpha]_D^{15} - 17·6^\circ$  in  $EtOH$ .

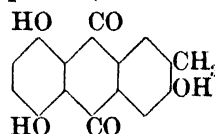
$B, HNO_3, \frac{1}{2} EtOH$ : m.p. 145°.

*Phenylurethane*: m.p. 200–2°.

*Methiodide*: m.p. 260°.

Barger, Seshadri, Watt, Yabuta, *J. Chem. Soc.*, 1935, 11.

### Rhababerone (Isoemodin, 3 : 5 : 8-trihydroxy-2-methylanthraquinone)



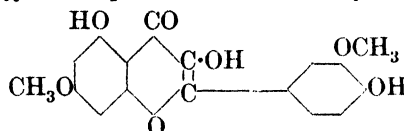
$C_{15}H_{10}O_5$

MW, 270

Occurs in rhubarb. Yellow leaflets from hot  $EtOH$ . M.p. 223–4° (212°). Sol. hot.  $EtOH$ , hot  $AcOH$ . Spar. sol.  $Et_2O$ . Insol.  $H_2O$ .  $FeCl_3 \rightarrow$  dark brown col. in  $EtOH$ . Sol. alkalis  $\rightarrow$  purple-red col.

Keimatsu, Hirano, *Chem. Abstracts*, 1931, **25**, 3647; *Chem. Zentr.*, 1931, I, 3348.

### Rhamnazin (3 : 5 : 4'-Trihydroxy-7 : 3'-dimethoxyflavone, quercetin 7 : 3'-dimethyl ether)



$C_{17}H_{14}O_7$

MW, 330

Found in fruits of *Rhamnus infectoria*, Linn. Pale yellow needles from toluene. M.p. 214–15°. Mod. sol. boiling  $AcOH$ , toluene. Spar. sol.  $EtOH$ . Alkalis  $\rightarrow$  orange-red sols.  $FeCl_3 \rightarrow$  olive-green col.

*Triacetyl*: needles from  $EtOH$ . M.p. 154–5°.

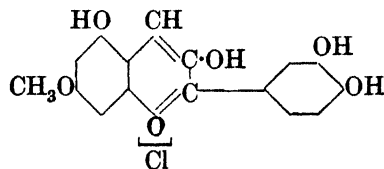
*Tribenzoyl*: needles from  $AcOH$ . M.p. 204–5°. Spar. sol.  $AcOH$ .

Perkin, Allison, *J. Chem. Soc.*, 1902, **81**, 469.

### $\alpha$ -Rhamnegin.

See Xanthorhamnin.

### Rhamnetidin chloride



$C_{16}H_{13}O_6Cl$

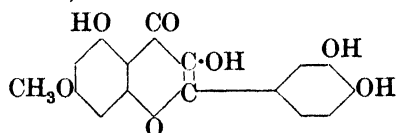
MW, 336·5

Purplish-brown prisms +  $2H_2O$ . Orange-red by transmitted light. Green reflex. Sol.  $EtOH \rightarrow$  bluish-red col. Sol. hot  $HCl \rightarrow$  brownish-red col.  $Na_2CO_3 \rightarrow$  blue col.  $FeCl_3 \rightarrow$  blue col. in  $EtOH$ , violet in  $H_2O$ .

Kondo, Segawa, *Chem. Abstracts*, 1932, **26**, 4333.

Robertson, Robinson, *J. Chem. Soc.*, 1927, 2205.

**Rhamnetin** (*Quercetin 7-methyl ether*, 3 : 5 : 3' : 4'-tetrahydroxy-7-methoxyflavone,  $\beta$ -rhamnocitrin)



$C_{16}H_{12}O_7$

MW, 316

Occurs in fruit of *Rhamnus cathartica*, Linn., and other species. Yellow needles from EtOH or phenol. Does not melt below 300°. Sol.  $Me_2CO$ , hot EtOH. Spar. sol.  $H_2O$ .  $FeCl_3 \rightarrow$  brown col. Conc.  $H_2SO_4 \rightarrow$  yellow col. with greenish-blue fluor. Reduces cold Tollen's and warm Fehling's.

*Tetra-acetyl*: m.p. 190–2°.

*Tetra-propionyl*: m.p. 158–62°.

Herzig, *Monatsh.*, 1888, **9**, 549.

Liebermann, Hörmann, *Ann.*, 1879, **196**, 313.

Krassowski, *Chem. Zentr.*, 1909, **I**, 772.

Oesch, Perkin, *J. Chem. Soc.*, 1914, **105**, 2354.

### Rhamninose

$C_{18}H_{32}O_{14}$

MW, 472

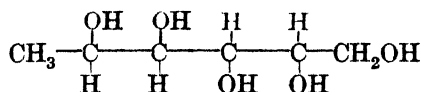
Obtained from xanthorhamnin present in Persian berries (*Rhamnus infectoria*). Cryst. M.p. 135–40° decomp. Sweet taste.  $[\alpha]_D - 41.0^\circ$  in  $H_2O$ ,  $- 26.37^\circ$  in 75% EtOH. Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ ,  $Me_2CO$ , AcOEt. Reduces Fehling's. Hyd. by dil. acids  $\rightarrow$  2 mols. rhamnose + 1 mol. galactose.  $NaHg \rightarrow$  rhamninitol. Br water  $\rightarrow$  rhamnotrionic acid.

*Octa-acetyl*: cryst. M.p. 95°.  $[\alpha]_D - 30.87^\circ$  in EtOH,  $- 31.7^\circ$  in AcOH.

Ter Meulen, *Rec. trav. chim.*, 1923, **42**, 380.

Tanret, Tanret, *Bull. soc. chim.*, 1899, **21**, 1065.

### Rhamnitol



$C_6H_{14}O_5$

MW, 166

*d.*

Prisms from  $Me_2CO$ . M.p. 123°. Sol.  $H_2O$ , EtOH. Spar. sol.  $CHCl_3$ . Insol.  $Et_2O$ .  $[\alpha]_D^{20} - 12.4^\circ$  in  $H_2O$ .

*Dibenzylidene deriv.*: m.p. 207°.  $[\alpha]_D + 60.7^\circ$  in  $CHCl_3$ .

*l.*

M.p. 121°.  $[\alpha]_D^{20} + 10.7^\circ$  in  $H_2O$ .

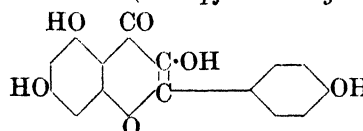
*Dibenzylidene deriv.*: cryst. M.p. 203°.  $[\alpha]_D - 55^\circ$  in  $CHCl_3$ .

Votoček, Valentin, *Rac. Chem. Abstracts*, 1931, **25**, 84.

Fischer, Piloty, *Ber.*, 1890, **23**, 3103.

de Bruyn, van Ekenstein, *Rec. trav. chim.*, 1899, **18**, 151.

### Rhamnocitrin (*Kaempferol methyl ether*)



One of the OH groups is methylated

$C_{16}H_{12}O_6$

MW, 300

Occurs in fruits of *Rhamnus cathartica*, Linn. Yellow needles. M.p. 221–2°. Mod. sol.  $Me_2CO$ , AcOEt. Spar. sol. hot EtOH. Insol.  $H_2O$ ,  $Et_2O$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  green fluor. Reduces Fehling's.

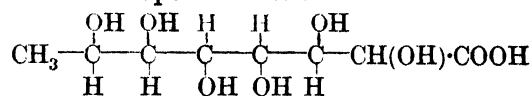
*Triacetyl*: m.p. 200–1°.

Oesch, Perkin, *J. Chem. Soc.*, 1914, **105**, 2352.

### $\beta$ -Rhamnocitrin.

See Rhamnetin.

### Rhamnoheptonic Acid



$C_8H_{16}O_8$

MW, 240

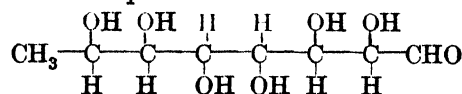
Free acid not isolated.

$\gamma$ -Lactone:  $C_8H_{14}O_7$ . MW, 222. Needles from EtOH. M.p. 160°.  $[\alpha]_D^{20} + 55.6^\circ$  in  $H_2O$ . Sol.  $H_2O$ . Mod. sol. MeOH, EtOH. Insol.  $Et_2O$ .

*Phenylhydrazide*: needles from  $H_2O$ . M.p. about 215° decomp. Spar. sol.  $H_2O$ , EtOH.

Fischer, Piloty, *Ber.*, 1890, **23**, 3106.

### Rhamnoheptose



$C_8H_{16}O_7$

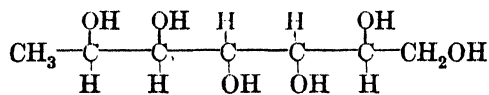
MW, 224

Syrup. Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .  $[\alpha]_D^{20} + 8.4^\circ$  in  $H_2O$ .

*Phenylosazone*: yellow needles. M.p. 200° decomp.

Anderson, *J. Am. Chem. Soc.*, 1911, **33**, 1514.

Fischer, Piloty, *Ber.*, 1890, **23**, 3107.

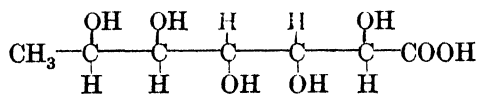
$\alpha$ -Rhamnohexitol $\text{C}_7\text{H}_{16}\text{O}_6$ 

MW, 196

Prisms from EtOH. M.p. 173°.  $[\alpha]_D^{20} + 14^\circ$  in  $\text{H}_2\text{O}$ . Mod. sol. hot MeOH, hot EtOH.

Fischer, Piloty, *Ber.*, 1890, **23**, 3106.

Valentin, *Chem. Abstracts*, 1932, **26**, 1578.

 $\alpha$ -Rhamnohexonic Acid $\text{C}_7\text{H}_{14}\text{O}_7$ 

MW, 210

M.p. 171°.  $[\alpha]_D^{20} + 21.5^\circ$  in  $\text{H}_2\text{O}$ .

$\text{NH}_4$  salt: m.p. 151°.

Brucine salt: m.p. 120–3°.

Et ester:  $\text{C}_9\text{H}_{18}\text{O}_7$ . MW, 238. M.p. 165–6° decomp.  $[\alpha]_D^{20} + 12.9^\circ$  in  $\text{H}_2\text{O}$ .

Amide:  $\text{C}_7\text{H}_{15}\text{O}_6\text{N}$ . MW, 209. M.p. 177.5–178° decomp. (194°).  $[\alpha]_D^{20} - 19.9^\circ$  in  $\text{H}_2\text{O}$  (–47.26°).

Nitrile:  $\text{C}_7\text{H}_{13}\text{O}_5\text{N}$ . MW, 191. M.p. 145°.  $[\alpha]_D^{20} - 23.47^\circ$ .

$\gamma$ -Lactone:  $\text{C}_7\text{H}_{12}\text{O}_6$ . MW, 192. Cryst. from  $\text{Me}_2\text{CO}$ . M.p. 171–171.5° (174.5°).  $[\alpha]_D^{20} + 87.3^\circ$  in  $\text{H}_2\text{O}$ .

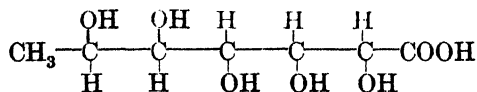
Phenylhydrazide: m.p. 205–6° decomp.  $[\alpha]_D^{20} - 5.2^\circ$  in  $\text{H}_2\text{O}$ .

Brackenbury, Upson, *J. Am. Chem. Soc.*, 1934, **56**, 2659.

Jackson, Hudson, *ibid.*, 2455.

Miksic, *Chem. Abstracts*, 1929, **23**, 2942.

Fischer, Morrell, *Ber.*, 1894, **27**, 386.

 $\beta$ -Rhamnohexonic Acid $\text{C}_7\text{H}_{14}\text{O}_7$ 

MW, 210

Free acid not isolated.

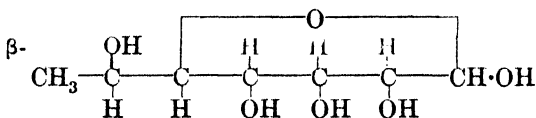
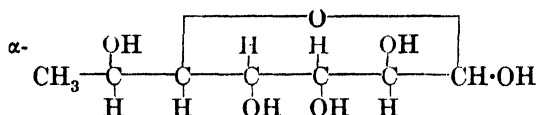
Brucine salt: m.p. 114–18°.

$\gamma$ -Lactone: plates from  $\text{Me}_2\text{CO}$ . M.p. 134–8°.  $[\alpha]_D^{20} + 43.34^\circ$  in  $\text{H}_2\text{O}$ .

Phenylhydrazide: m.p. 170° decomp.

Fischer, Morrell, *Ber.*, 1894, **27**, 389.

## Rhamnohexose

 $\text{C}_7\text{H}_{14}\text{O}_6$ 

MW, 194

 $\alpha$ -*l*-

Prisms or plates from hot MeOH. M.p. 180–1°. Sol.  $\text{H}_2\text{O}$ , hot MeOH. Spar. sol. EtOH.  $[\alpha]_D^{20} - 80^\circ \rightarrow -61.4^\circ$  in  $\text{H}_2\text{O}$ . Sweet taste.  $\text{NaHg} \rightarrow \alpha$ -rhamnohexitol.

Phenylosazone: yellow needles. M.p. 200° decomp. Sol. hot EtOH. Insol.  $\text{H}_2\text{O}$ .

Benzylphenylhydrazone: m.p. 183–4°.

 $\beta$ -*l*-

Syrup.

Phenylosazone: identical with that of  $\alpha$ -form.

Monobenzylidene deriv.: m.p. 233–4°.  $[\alpha]_D^{20} + 50.8^\circ$  in  $\text{CHCl}_3$ .

Votoček, Valentin, Leminger, *Chem. Abstracts*, 1931, **25**, 4527.

Votoček, Valentin, Rac, *ibid.*, 84.

Anderson, *J. Am. Chem. Soc.*, 1911, **33**, 1514.

Fischer, Morrell, *Ber.*, 1894, **27**, 391.

Rhamnol (*Cinchol*) $\text{C}_{29}\text{H}_{50}\text{O}$ 

MW, 414

Phytosterol occurring in cascara and other barks. M.p. 136–7°.  $[\alpha]_D^{18} - 33.5^\circ$  in  $\text{CHCl}_3$ .

Acetyl deriv.: m.p. 123°.  $[\alpha]_D^{18} - 38.3^\circ$  in  $\text{CHCl}_3$ .

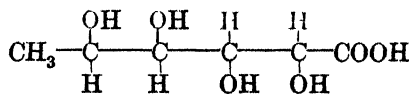
3 : 5-Dinitrobenzoyl deriv.: m.p. 200–2°.  $[\alpha]_D^{18} - 10.9^\circ$  in  $\text{CHCl}_3$ .

Windaus, Deppe, *Ber.*, 1933, **66**, 1689.

## Rhamnolutin.

See Kämpferol.

## Rhamnonic Acid

 $\text{C}_6\text{H}_{12}\text{O}_6$ 

MW, 180

*l*-

Obtained only in solution.  $[\alpha]_D - 7.67 \rightarrow 29.28^\circ$  in  $\text{H}_2\text{O}$ .

*Brucine salt*: m.p. 120–6° (132°).

*Quinine salt*: m.p. 180–1°.

*Nitrile: tetra-acetyl deriv.*: m.p. 69–70°.

*γ-Lactone*:  $C_8H_{10}O_5$ . MW, 162. M.p. 149–51°.  $[\alpha]_D^{20} - 39.2^\circ$  in  $H_2O$ .

*δ-Lactone*: m.p. 178–82°.  $[\alpha]_D + 98-101^\circ$ .

*Amide*:  $C_8H_{13}O_5N$ . MW, 179. M.p. 134–134.5°.  $[\alpha]_D^{20} + 27.7^\circ$  in  $H_2O$ .

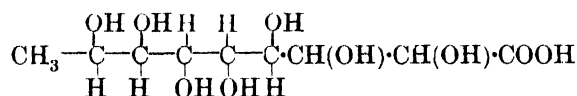
*Phenylhydrazide*: m.p. 195–6°.  $[\alpha]_D^{80} + 17.2^\circ$  in  $H_2O$ .

Brackenbury, Upson, *J. Am. Chem. Soc.*, 1933, **55**, 2514.

Isbell, Frush, *Chem. Abstracts*, 1934, **28**, 1667.

Rehorst, *Ann.*, 1933, **503**, 143.

### Rhamno-octonic Acid



$C_9H_{18}O_9$

MW, 270

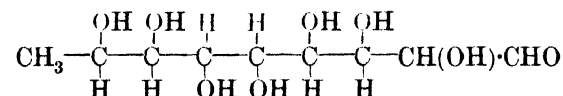
Free acid not isolated.

*γ-Lactone*:  $C_9H_{16}O_8$ . MW, 252. Needles from  $H_2O$ . M.p. 171–2°.  $[\alpha]_D^{20} - 50.8^\circ$  in  $H_2O$ . Sol.  $H_2O$ , EtOH. Spar. sol.  $Me_2CO$ . NaHg  $\rightarrow$  rhamno-octose.

*Phenylhydrazide*: needles from  $H_2O$ . M.p. about 220° decomp. Spar. sol. hot  $H_2O$ .

Fischer, Piloty, *Ber.*, 1890, **23**, 3109, 3827.

### Rhamno-octose



$C_9H_{18}O_8$

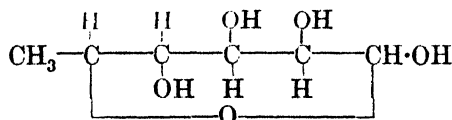
MW, 254

Syrup. Sol.  $H_2O$ .

*Phenylosazone*: m.p. 216°.

Fischer, Piloty, *Ber.*, 1890, **23**, 3110.

### Rhamnose



$C_6H_{12}O_5$

MW, 164

*d.*

Cryst. +  $H_2O$ . Sol.  $H_2O$ . Insol.  $Et_2O$ .  $[\alpha]_D^{16.5} - 8.25^\circ$  in  $H_2O$ . Reduces warm Fehling's. NaHg  $\rightarrow$  *d*-rhamnitol.

*Phenylosazone*: yellow prisms or needles from dil. EtOH. M.p. 191° (185°, 186–7°, 189–90°).  $[\alpha]_D^{20} - 95.2^\circ$  in Py. Sol. EtOH, Py. Identical

with phenylosazone of *d*-isorhodeose and *d*-isorhamnose.

*p*-Bromophenylosazone: yellow cryst. from dil. EtOH. M.p. 225° (222–3°).

*l.*

*α-Form*:

A constituent of many glucosides. Cryst. +  $H_2O$  from  $H_2O$ . M.p. 105° (93–4°). Sweet taste.  $[\alpha]_D^{15} + 9.1^\circ$  in  $H_2O$  ( $[\alpha]_D^{20} - 7.7^\circ \rightarrow + 8.9^\circ$  in  $H_2O$ ). Sol.  $H_2O$ , MeOH. Reduces warm Fehling's.

*Oxime*: plates. M.p. 127–8°.  $[\alpha]_D^{20} + 13.70^\circ$  in  $H_2O$  (final). Sol.  $H_2O$ . Spar. sol. EtOH. Insol.  $Et_2O$ .

*Semicarbazone*: cryst. from dil. EtOH. M.p. 183°.  $[\alpha]_D^{20} + 75^\circ \rightarrow + 57^\circ$  in  $H_2O$ .

*Phenylhydrazone*: laminæ. M.p. 159°.  $[\alpha]_D^{20} + 54.2^\circ$  in  $H_2O$ ,  $[\alpha]_D + 27^\circ$  in EtOH. Mod. sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .

*Diphenylhydrazone*: prisms. M.p. 134°. Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .

*p*-Nitrophenylhydrazone: reddish-yellow cryst. M.p. 186° (191–2°).  $[\alpha]_D + 21.4^\circ$  (– 50.3°  $\rightarrow$  – 8.5°) in EtOH–Py. Sol. EtOH.

*Methylphenylhydrazone*: cryst. M.p. 124°.  $[\alpha]_D + 0.7^\circ$  in MeOH. Sol. MeOH. Spar. sol.  $H_2O$ , EtOH.

*Benzylphenylhydrazone*: yellow cryst. M.p. 121°.  $[\alpha]_D - 6.4^\circ$  in MeOH.

*β*-Naphthylhydrazone: brown needles. M.p. 170°.  $[\alpha]_D + 8.4^\circ$  in MeOH, – 11.8° in AcOH. Sol. MeOH. Spar. sol.  $H_2O$ .

*Phenylosazone*: yellow needles. M.p. 222° (180°, 185°, 186–7°, 182°).  $[\alpha]_D^{20} + 93.92^\circ$  (94°) in Py. Sol. hot EtOH, AcOH,  $Me_2CO$ . Spar. sol. EtOH,  $C_6H_6$ . Insol.  $H_2O$ . Reduces boiling Fehling's.

*p*-Nitrophenylosazone: red needles from EtOH. M.p. 208° decomp. Spar. sol. EtOH.

*Methylglucoside*: cryst. from AcOEt. M.p. 108–9° (109–10°).  $[\alpha]_D^{20} - 62.5^\circ$  (– 25.43°) in  $H_2O$ . *Tri-Me ether*: b.p. 112°/11 mm., 101°/9 mm.  $[\alpha]_D^{20} - 15.54^\circ$  in  $H_2O$ . *Triacetyl*: m.p. 86–7°.  $[\alpha]_D^{16} - 53.5^\circ$  in  $C_2H_2Cl_4$ .

*2:3:4-Tri-Me ether*: syrup. B.p. 141°/19 mm.  $n_D^{20} 1.4565$ .  $[\alpha]_D^{18} + 24.15^\circ \rightarrow + 25.44^\circ$  in  $H_2O$ , + 3.25°  $\rightarrow$  + 5.82° in  $C_6H_6$ , – 4.86°  $\rightarrow$  – 9.52° in EtOH. *Phenylhydrazone*: yellow prisms. M.p. 126–8° decomp.

*Tetranitrate*: cryst. from EtOH. M.p. 135° decomp.

*β-Form*:

Needles from  $Me_2CO$ . M.p. 122–6°.  $[\alpha]_D + 54.0^\circ \rightarrow + 8.9^\circ$  in  $H_2O$ . Sol.  $H_2O$ , EtOH.

*Methylglucoside*: needles from AcOEt. M.p.



138–40°.  $[\alpha]_D^{20} + 95.39^\circ$  in  $H_2O$ . *Triacetyl*: m.p. 151–2°.  $[\alpha]_D^{18} + 45.73^\circ$  in  $C_2H_2Cl_4$ . *Triacetyl*: plates from  $Et_2O$ . M.p. 96–8°.  $[\alpha]_D^{21} + 28.09^\circ \longrightarrow + 18.6^\circ$  in  $EtOH$ .

*dl*–.

M.p. anhyd. 151.3–153°.

Votoček, Valentin, *Rac. Chem. Abstracts* 1931, **25**, 84.

Votoček, Valentin, *Compt. rend.*, 1926, **183**, 62.

Fischer, Zach, *Ber.*, 1912, **45**, 3770.

Harding, *Sugar*, 1923, **25**, 82.

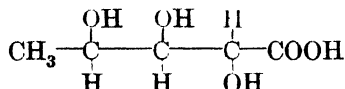
Hirst, Macbeth, *J. Chem. Soc.*, 1926, 22.

Clark, *J. Biol. Chem.*, 1919, **38**, 255.

Walton, *J. Am. Chem. Soc.*, 1921, **43**, 127.

Fischer, Bergmann, Rabe, *Ber.*, 1920, **53**, 2362.

### Rhamnotetronic Acid (*Methyltetronic acid*)



$C_5H_{10}O_5$

MW, 150

*l*–.

Free acid not isolated.

*Brucine salt*: needles from  $EtOH$ . M.p. 145–50° decomp.

*Amide*:  $C_5H_{11}O_4N$ . MW, 149. Plates from  $EtOH$ . M.p. 135° decomp.  $[\alpha]_D + 54.8^\circ$  in  $H_2O$ .

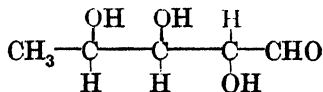
*Phenylhydrazide*: laminæ from  $AcOEt$ . M.p. 169°.

$\gamma$ -Lactone:  $C_5H_8O_4$ . MW, 132. Needles from  $H_2O$  or  $EtOH$ . M.p. 123° (120–1°).  $[\alpha]_D^{20} - 47.5^\circ$  in  $H_2O$ . Sol.  $EtOH$ ,  $AcOEt$ ,  $Et_2O$ . Spar. sol.  $CHCl_3$ ,  $C_6H_6$ .

Ruff, *Ber.*, 1902, **35**, 2365.

Hudson, Chernoff, *J. Am. Chem. Soc.*, 1918, **40**, 1005.

### Rhamnotetrose (*Methyltetrose*)



$C_5H_{10}O_4$

MW, 134

*l*–.

Syrup. Sol.  $H_2O$ ,  $EtOH$ .  $[\alpha]_D^{20} - 30.5^\circ \longrightarrow - 16.35^\circ$  in 96%  $EtOH$ . Reduces Fehling's.  $HNO_3 \longrightarrow$  tartaric acid.

*Benzylphenylhydrazone*: needles from  $C_6H_6$ . M.p. 96–7°.  $[\alpha]_D^{20} - 6.5^\circ$  in 96%  $EtOH$ . Sol.  $EtOH$ ,  $Et_2O$ . Prac. insol.  $H_2O$ ,  $C_6H_6$ .

*Phenylosazone*: yellow needles from  $EtOH$ .

M.p. 172–3°. Sol. hot  $EtOH$ ,  $C_6H_6$ . Prac. insol.  $H_2O$ ,  $Et_2O$ .

Ruff, *Ber.*, 1902, **35**, 2364.

Fischer, *Ber.*, 1896, **29**, 1381.

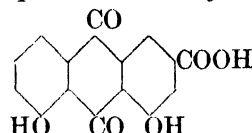
### Rhamnnoxanthin.

Frangulin, *q.v.*

### Rheic Acid.

Chrysophanic Acid, *q.v.*

**Rhein** (*Chrysazin-3-carboxylic acid*, 4:5-dihydroxyanthraquinone-2-carboxylic acid)



$C_{15}H_8O_6$

MW, 284

Occurs in senna leaves. Yellow needles from  $MeOH$ . M.p. 321°. Sol. Py. Spar. sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ ,  $AcOH$ ,  $Me_2CO$ ,  $CHCl_3$ , pet. ether.

*Diacetyl*: m.p. 258° (246° decomp.).

*Mono-propionyl deriv.*: m.p. 223–4°. *Me ether*: m.p. 281–2°.

*Dipropionyl*: m.p. 223–4°.

*Dibenzoyl*: m.p. 262° (253–5°).

*Me ester*:  $C_{16}H_{10}O_6$ . MW, 298. Orange needles from  $MeOH$ . M.p. 174°.

*Et ester*:  $C_{17}H_{12}O_6$ . MW, 312. Orange needles from  $EtOH$ . M.p. 159° (160–1°).

*Diacetyl*: m.p. 170°.

*Propyl ester*:  $C_{18}H_{14}O_6$ . MW, 326. Brown needles. M.p. 145°. *Diacetyl*: m.p. 178°.

*Isopropyl ester*: brown cryst. M.p. 181°. *Diacetyl*: m.p. 190°.

*Isobutyl ester*:  $C_{19}H_{16}O_6$ . MW, 340. Yellow needles. M.p. 153°. *Diacetyl*: m.p. 109°.

*Phenyl ester*:  $C_{21}H_{12}O_6$ . MW, 360. Yellow needles. M.p. 215°. *Diacetyl*: m.p. 170°.

*Benzyl ester*:  $C_{22}H_{20}O_6$ . MW, 374. *Diacetyl*: m.p. 203°.

*Di-Me ether*:  $C_{17}H_{12}O_6$ . MW, 312. Brown needles from  $EtOH$ . M.p. 283–4°. *Et ester*:  $C_{19}H_{16}O_6$ . MW, 340. Yellow needles from  $AcOH$ . M.p. 185–7°. *Chloride*:  $C_{17}H_{11}O_5Cl$ . MW, 330.5. Yellow prisms from  $CHCl_3$ -pet. ether. M.p. 190°. *Amide*:  $C_{17}H_{13}O_5N$ . MW, 311. Brownish-yellow plates from  $AcOEt$ . M.p. 287°.

Oesterle, Haugseth, *Arch. Pharm.*, 1915, **253**, 330.

### Rheochrysidine.

See *Physcione*.

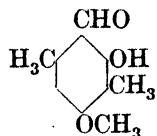
### Rheumatin.

See under *Quinine*.

### Rheum-emodin.

Frangula-emodin, *q.v.*

**Rhizonaldehyde** (2-Hydroxy-4-methoxy-3:6-dimethylbenzaldehyde,  $\beta$ -orcylaldehyde 5-methyl ether)



$C_{10}H_{12}O_3$  MW, 180

Prisms from EtOH.Aq. M.p. 136°. Sol.  $Et_2O$ ,  $C_6H_6$ , hot EtOH, hot pet. ether.  $FeCl_3$  → reddish-brown col.

Oxime: m.p. 188-9°.

Acetyl: m.p. 71°.

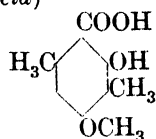
Carbomethoxyl: m.p. 90°.

Sonn, *Ber.*, 1916, **49**, 2591.

Robertson, Stephenson, *J. Chem. Soc.*, 1930, 318.

Pfau, *Helv. Chim. Acta*, 1928, **11**, 873.

**Rhizonic Acid** (2-Hydroxy-4-methoxy-3:6-dimethylbenzoic acid)



$C_{10}H_{12}O_4$  MW, 196

Occurs in *Evernii prunastri*, and *Rizocarpon geographicum*, Linn. Cryst. from EtOH. M.p. 235° (232°).

Me ester:  $C_{11}H_{14}O_4$ . MW, 210. Needles from EtOH. M.p. 95°. Me ether:  $C_{12}H_{16}O_4$ . MW, 224. B.p. 161-3°/10 mm.

Et ester:  $C_{12}H_{16}O_4$ . MW, 224. Needles from EtOH. M.p. 82° (81°).

Acetyl: m.p. 146° decomp.

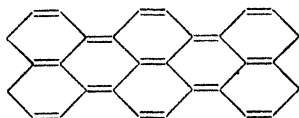
2-Me ether:  $C_{11}H_{14}O_4$ . MW, 210. Prisms from pet. ether. M.p. 104-5°.

Asahina, Akagi, *Ber.*, 1935, **68**, 1132.

Robertson, Stephenson, *J. Chem. Soc.*, 1930, 319.

Sonn, *Ber.*, 1929, **62**, 3012.

**Rhodacene** (Quinonoid form of chalkacene, q.v.)



$C_{30}H_{16}$  MW, 376

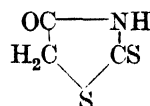
Violet microcryst. M.p. 338-40°. Sol.  $C_6H_6$  and homologues with red col. and red fluor.

Dziewoński, Podgorska, Lemberger, Suszka, *Ber.*, 1920, **53**, 2173.

**Rhodanic Acid.**

See Rhodanine.

**Rhodanine** (Rhodanic acid)



$C_3H_3ONS_2$  MW, 133

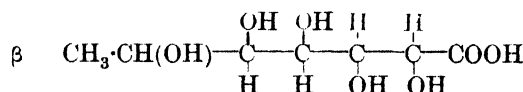
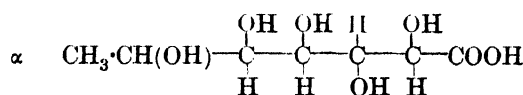
Pale yellow prisms from EtOH. M.p. 170°.

Julian, Sturgis, *J. Am. Chem. Soc.*, 1935, **57**, 1126.

**Rhodeitol.**

See Fucitol.

**Rhodeohexonic Acid**



$C_7H_{14}O_7$  MW, 210

$\alpha$ -Form:

Syrup rapidly changing to lactone.

$\gamma$ -Lactone:  $C_7H_{12}O_6$ . MW, 192. Cryst. +  $2H_2O$ . M.p. 41-5° (129-31°).

Amide:  $C_7H_{15}O_6N$ . MW, 209. M.p. 206°.

Phenylhydrazide: m.p. 231° decomp.

$\beta$ -Form:

Syrup rapidly changing to lactone.

$\gamma$ -Lactone: m.p. 115°.

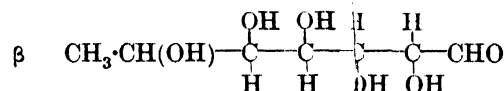
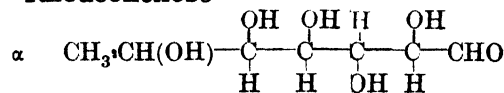
Amide: amorph. grey powder. M.p. 197-8°.

Phenylhydrazide: m.p. 211° decomp.

Votoček, *Chem. Abstracts*, 1935, **29**, 2918; *Ber.*, 1910, **43**, 469.

Krauz, *Ber.*, 1910, **43**, 482.

**Rhodeohexose**



$C_7H_{14}O_6$  MW, 194

$\alpha$ -Form:

Cryst. from EtOH. M.p. 125-6°.  $[\alpha]_D^{20} + 11.96^\circ$  in  $H_2O$ .

Phenylhydrazone: m.p. 150°.

p-Bromophenylhydrazone: m.p. 173°.

$\beta$ -Form :

Syrup.

*Phenylhydrazone* : m.p. 131-7°.*p-Bromophenylhydrazone* : m.p. 145°.Krauz, *Chem. Zentr.*, 1911, II, 1216;  
*Ber.*, 1910, 43, 482.**Rhodeoretin.**

See Convolvulin.

**Rhodoese.**

See Fucose.

**Rhodinal.**

See Citronellal.

**Rhodinic Acid.**

See Citronellic Acid.

**Rhodinol.**

See Citronellol.

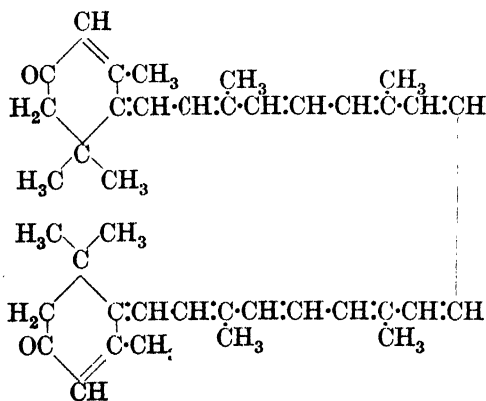
**Rhodinolic Acid.**

See Citronellic Acid.

**Rhodoviolascin**

$$\text{C}_{42}\text{H}_{60}\text{O}_2 \quad \text{C}_{40}\text{H}_{54}(\text{OCH}_3)_2 \quad \text{MW, 596}$$

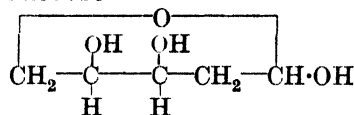
Carotinoid pigment from *Rhodovibrio* bacteria. Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 218°. Contains 13 double bonds.

Karrer, Solmssen, *Helv. Chim. Acta*, 1936, 19, 3.**Rhodoxanthin**

$$\text{C}_{40}\text{H}_{50}\text{O}_2 \quad \text{MW, 562}$$

Pigment occurring in *Taxus baccata*, Linn. (Yew). Bluish-black leaflets from  $\text{C}_6\text{H}_6$ -MeOH. M.p. 219°. Absorption maxima in  $\text{CS}_2$  at 564, 525 and 491 m $\mu$ .

*Dioxime* : m.p. 227-8°. Absorption maxima in  $\text{CS}_2$  at 516, 483 and 453 m $\mu$ .

Kuhn, Frockmann, *Ber.*, 1933, 66, 828.**2-Ribodesose**

$$\text{C}_5\text{H}_{10}\text{O}_4 \quad \text{MW, 134}$$

Cryst. from propyl alcohol. M.p. 90°. Sol.  $\text{H}_2\text{O}$ .  $[\alpha]_D^{25} + 2.13^\circ$  in Py,  $+ 2.88^\circ \rightarrow + 2.13^\circ$  in  $\text{H}_2\text{O}$ . Reduces Fehling's. Sweet taste.

*Benzylphenylhydrazone* : m.p. 127-9°.

There are described also :

*d*-2-Ribodesose (Thyminosose).

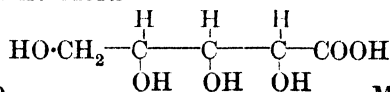
Cryst. M.p. 78°.  $[\alpha]_D^{25} - 90.6^\circ \rightarrow 40.0^\circ$  in Py,  $- 60^\circ \rightarrow - 50^\circ$  in  $\text{H}_2\text{O}$ .

*Benzylphenylhydrazone* : cryst. M.p. 128°.  $[\alpha]_D^{25} - 17.5^\circ$  in Py.

*l*-2-Ribodesose (*l*-Arabodesose).

Cryst. M.p. 80°.  $[\alpha]_D^{25} + 91.7^\circ \rightarrow + 40.5^\circ$  in Py.

*Benzylphenylhydrazone* : m.p. 125-6°.  $[\alpha]_D^{25} + 17.5^\circ$  in Py.

Meisenheimer, Jung, *Ber.*, 1927, 60, 1464.Levene, Mori, *J. Biol. Chem.*, 1929, 83, 803.Levene, Mikeska, Mori, *J. Biol. Chem.*, 1930, 85, 785.**Ribonic Acid**

$$\text{C}_5\text{H}_{10}\text{O}_6 \quad \text{MW, 166}$$
*d*-.

Obtained only in solution.  $[\alpha]_D + 8.42^\circ$  in  $\text{H}_2\text{O}$  (final).

$\gamma$ -Lactone :  $\text{C}_5\text{H}_8\text{O}_5$ . MW, 148. Cryst. from AcOEt. M.p. 72-8° (80°).  $[\alpha]_D + 18.4^\circ$  in  $\text{H}_2\text{O}$ .

*Hydrazide* : m.p. 150°.  $[\alpha]_D^{15} + 27.5^\circ$ .*l*-.

M.p. 104-5°.  $[\alpha]_D^{20} + 17.6^\circ$  in  $\text{H}_2\text{O}$ .

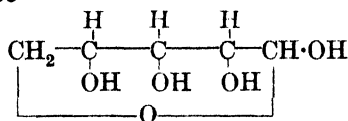
$\gamma$ -Lactone : prisms from AcOEt. M.p. 84-6° (80°, 72-6°). Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Me}_2\text{CO}$ . Mod. sol. AcOEt. Spar. sol. Et<sub>2</sub>O.  $[\alpha]_D^{20} - 18^\circ$  in  $\text{H}_2\text{O}$ .

*Amide* :  $\text{C}_5\text{H}_{11}\text{O}_5\text{N}$ . MW, 165. Plates. M.p. 136-7°.  $[\alpha]_D^{14} - 18.7^\circ$ .

*Phenylhydrazide* : m.p. 204°.  $[\alpha]_D^{18} - 33^\circ$  in  $\text{H}_2\text{O}$ .

Schmidt, Weber-Molster, *Ann.*, 1934, 515, 43.Rehorst, *Ann.*, 1933, 503, 143.Fischer, Piloty, *Ber.*, 1891, 24, 4216.Hasenfratz, *Compt. rend.*, 1927, 184, 210.v. Ekenstein, Blankensma, *Chem. Zentr.*, 1913, II, 1562.

## Ribose

 $\text{C}_5\text{H}_{10}\text{O}_5$ 

MW, 150

*d*-.  
A constituent of many nucleosides and nucleotides. Cryst. M.p. 95° (86–7°). Hygroscopic.  $[\alpha]_D - 21.5^\circ$  (–19.5°) in  $\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH.

*p*-Bromophenylhydrazone: cryst. M.p. 170° (166–7°).  $[\alpha]_D + 5.69^\circ$  in EtOH.

Phenylosazone: yellow cryst. from  $\text{H}_2\text{O}$ . M.p. 160° (162–3°, 163–4°). Identical with phenylosazone of *d*-arabinose.

*p*-Bromophenylosazone: m.p. 180–5°.

$\beta$ -Methylglucoside: m.p. 83–4°.  $[\alpha]_D^{20} - 113.6^\circ$ .

*l*-.  
Cryst. from EtOH. M.p. 87°.  $[\alpha]_D + 18.8^\circ$  in  $\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Sweet taste.

Phenylhydrazone: cryst. from EtOH. M.p. 154–5° decomp. Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH.

*p*-Bromophenylhydrazone: cryst. from EtOH. M.p. 164–5°. Sol.  $\text{H}_2\text{O}$ .

Phenylosazone: yellow cryst. from  $\text{Me}_2\text{CO}$  or  $\text{H}_2\text{O}$ . M.p. 166°. Identical with phenylosazone of *l*-arabinose.  $[\alpha]_D + 1^\circ$  in Py–EtOH, +18.9° in EtOH. Sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Me}_2\text{CO}$ , Py. Insol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin.

*dl*-.  
M.p. 83–4°.

Phenylosazone: yellow needles or prisms. M.p. 166–8°. Identical with phenylosazone of *dl*-arabinose.

Austin, Humoller, *J. Am. Chem. Soc.*, 1934, **56**, 1152.

Levene, *J. Biol. Chem.*, 1935, **108**, 419.

Steiger, *Helv. Chim. Acta*, 1936, **19**, 189.

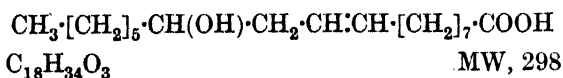
Fischer, Piloty, *Ber.*, 1891, **24**, 4220.

Levene, Jacobs, *Ber.*, 1909, **42**, 3247.

v. Ekenstein, Blankema, *Chem. Zentr.*, 1913, II, 1562; 1914, I, 965.

Minsaas, *Ann.*, 1934, **512**, 286.

**Ricinelaic Acid** (*Stereoisomer of ricinoleic acid*)

 $\text{C}_{18}\text{H}_{34}\text{O}_3$ 

MW, 298

Needles from ligroin. M.p. 51–2°. B.p. 240–2°/10 mm. Sol. EtOH.  $[\alpha]_D^{20} + 6.67^\circ$  in EtOH.

*Et ester*:  $\text{C}_{20}\text{H}_{38}\text{O}_3$ . MW, 326. M.p. 16°.

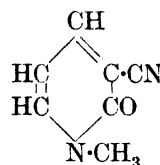
*Amide*:  $\text{C}_{18}\text{H}_{35}\text{O}_2\text{N}$ . MW, 297. M.p. 91–3°.

*Phenylhydrazone*: m.p. 110–110.5°.

Mühle, *Ber.*, 1913, **46**, 2096.

Krafft, *Ber.*, 1888, **21**, 2735.

## Ricinidine

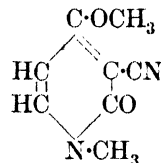
 $\text{C}_7\text{H}_6\text{ON}_2$ 

MW, 134

M.p. 140°. B.p. 243°/28 mm.

Späth, Koller, *Ber.*, 1923, **56**, 886.

**Ricinine** (4-Methoxy-2-keto-1-methyl-1:2-dihydronicotinonitrile, 4-methoxy-N-methyl-3-cyano-2-pyridone)

 $\text{C}_8\text{H}_8\text{O}_2\text{N}_2$ 

MW, 164

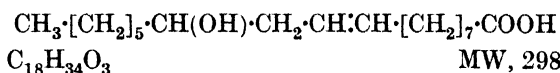
Occurs in castor-oil seeds. Leaflets or prisms from  $\text{H}_2\text{O}$ . M.p. 201.5°. Sol. hot  $\text{H}_2\text{O}$ , hot  $\text{CHCl}_3$ . Spar. sol. EtOH. Insol. pet. ether. Reduces  $\text{KMnO}_4$ .

*B, HgCl*<sub>2</sub>: m.p. 204°.

Späth, Koller, *Ber.*, 1925, **58**, 2124.

Schroeter, Seidler, Sulzbacher, Kametz, *Ber.*, 1932, **65**, 432.

Reitmann, *Chem. Abstracts*, 1935, **29**, 4359.

**Ricinoleic Acid** (*Ricinolic acid*) $\text{C}_{18}\text{H}_{34}\text{O}_3$ 

MW, 298

Occurs in castor oil as glyceride. B.p. 226–8°/10 mm. (245°/10 mm.). Sol. EtOH,  $\text{Et}_2\text{O}$ .  $[\alpha]_D^{25} + 5.05^\circ$ .  $D^{15} 0.9496$ .  $n_D^{15} 1.4145$ .

*Me ester*:  $\text{C}_{19}\text{H}_{36}\text{O}_3$ . MW, 312. B.p. 225–7°/15 mm. *Acetyl*: b.p. 210°/13 mm.  $D^{22} 0.9301$ .  $n_D^{22} 1.4570$ .  $[\alpha]_D^{22} + 15.25^\circ$ . *Propionyl*: b.p. 260°/13 mm.  $D^{18} 0.9226$ .  $n_D^{18} 1.4535$ .  $[\alpha]_D^{18} + 16.88^\circ$ . *Benzoyl*: b.p. 195–6°/0.08 mm.

*Et ester*:  $\text{C}_{20}\text{H}_{38}\text{O}_3$ . MW, 326. B.p. 258°/13 mm.  $D^{22} 0.9145$ .  $n_D^{22} 1.4618$ .  $[\alpha]_D^{22} + 5.28^\circ$ . *Acetyl*: b.p. 255–6°/13 mm.  $D^{22} 0.9170$ .  $n_D^{22} 1.4540$ .  $[\alpha]_D^{22} + 14.85^\circ$ . *Propionyl*: b.p. 265°/13 mm.  $D^{18} 0.9151$ .  $n_D^{18} 1.4517$ .  $[\alpha]_D^{18} + 16.06^\circ$ .

*Propyl ester*:  $C_{21}H_{40}O_3$ . MW, 340. B.p.  $268^\circ/13$  mm.  $D^{22}_D$  0.9079.  $n^{22}_D$  1.4573.  $[\alpha]^{22}_D + 4.15^\circ$ . *Acetyl*: b.p.  $260^\circ/13$  mm.  $D^{22}$  0.9117.  $n^{22}_D$  1.4513.  $[\alpha]^{22}_D + 14.40^\circ$ . *Propionyl*: b.p.  $310-20^\circ/645$  mm.  $D^{22}$  0.9128.  $n^{22}_D$  1.4498.  $[\alpha]^{22}_D + 13.61^\circ$ .

*Isopropyl ester*: b.p.  $210^\circ/10$  mm.  $D^{22}$  0.9083.  $n^{22}_D$  1.4583.  $[\alpha]^{22}_D + 4.04^\circ$ .

*Butyl ester*:  $C_{22}H_{42}O_3$ . MW, 354. B.p.  $278^\circ/13$  mm.  $D^{22}$  0.9058.  $n^{22}_D$  1.4566.  $[\alpha]^{22}_D + 3.73^\circ$ .

*Isobutyl ester*: b.p.  $282^\circ/9$  mm.  $D^{22}$  0.9028.  $n^{22}_D$  1.4538.  $[\alpha]^{22}_D + 4.01^\circ$ . *Acetyl*: b.p.  $255-60^\circ/13$  mm.  $D^{22}$  0.9012.  $n^{22}_D$  1.4548.  $[\alpha]^{22}_D + 9.58^\circ$ . *Propionyl*: b.p.  $325-35^\circ/160$  mm.  $D^{22}$  0.9027.  $n^{22}_D$  1.4525.  $[\alpha]^{22}_D + 9.45^\circ$ .

*Heptyl ester*:  $C_{25}H_{48}O_3$ . MW, 396. B.p.  $295^\circ/10$  mm.  $D^{22}$  0.8983.  $n^{22}_D$  1.4566.  $[\alpha]^{22}_D + 3.32^\circ$ .

*Phenylhydrazide*: m.p.  $63^\circ$ .

van Alphen, *Rec. trav. chim.*, 1925, **44**, 1064.

Ricler, U.S.P., 1,955,021, (*Chem. Abstracts*, 1934, **28**, 2926).

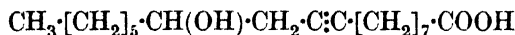
Straus, Heinze, Salzmann, *Ber.*, 1933, **66**, 631.

Böhme, D.R.P., 592,053, (*Chem. Abstracts*, 1934, **28**, 2725).

### Ricinolic Acid.

See Ricinoleic Acid.

### Ricinstearolic Acid



$C_{18}H_{32}O_3$  MW, 296

Needles from ligroin. M.p.  $52^\circ$ . B.p.  $260^\circ/10$  mm. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.  $[\alpha]_D + 13.67^\circ$  in H<sub>2</sub>O.

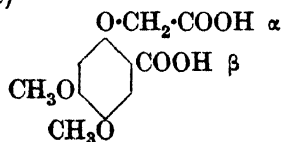
*A<sub>2</sub>Ba*: m.p.  $135^\circ$ .

*Me ester*:  $C_{19}H_{34}O_3$ . MW, 310. B.p.  $225^\circ/12$  mm.

*Et ester*:  $C_{20}H_{36}O_3$ . MW, 324. B.p.  $230^\circ/12$  mm.

Mühle, *Ber.*, 1913, **46**, 2091.

**Rissic Acid** (4 : 5-Dimethoxy-2-carboxyphenoxacetic acid)



$C_{11}H_{12}O_7$  MW, 256

Needles from hot butyl alcohol. M.p.  $262^\circ$  ( $256^\circ$  decomp.).

$\alpha$  :  $\beta$ -Di-Me ester:  $C_{13}H_{16}O_7$ . MW, 284. M.p.  $86^\circ$ . B.p.  $265-70^\circ/0.4$  mm.

$\alpha$ -Et ester:  $C_{13}H_{16}O_7$ . MW, 284. Prisms from EtOH. M.p.  $190^\circ$ .

Robertson, *J. Chem. Soc.*, 1932, 1380.

Butenandt, McCartney, *Ann.*, 1932, **494**, 17.

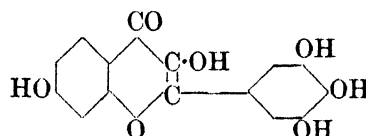
Clark, *J. Am. Chem. Soc.*, 1931, **53**, 2371; 1932, **54**, 2548.

Takei, Miyajima, Ono, *Ber.*, 1931, **64**, 251; *Chem. Abstracts*, 1932, **26**, 5300.

### Robigenin.

See Kämpferol.

**Robinetin** (3 : 7 : 3' : 4' : 5'-Pentahydroxyflavone)



$C_{15}H_{10}O_7$

MW, 302

Found in stem-wood of *Robinia pseudacacia*, Linn., and *Gleditsia monosperma*, Walt. Greenish-yellow needles from AcOH.Aq. Decomp. at  $325-30^\circ$ . Sol. EtOH, Me<sub>2</sub>CO, AcOH, AcOEt, Py. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether. Conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  yellow sol. Alkalis  $\rightarrow$  red sols. Drop of conc. H<sub>2</sub>SO<sub>4</sub> in red AcOH sol. gives characteristic intense green col.

3 : 3' : 4' : 5'-Tetra-Me ether:  $C_{19}H_{18}O_2$ . MW, 358. Pale yellow cryst. from EtOH. M.p.  $250-1^\circ$ . Alkalis and conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  yellow sols. *Acetyl*: needles from EtOH. M.p.  $149-50^\circ$ .

*Penta-Me ether*:  $C_{20}H_{20}O_7$ . MW, 372. Prisms from MeOH. M.p.  $149^\circ$ . Conc. HCl  $\rightarrow$  yellow sol.

*Penta-acetyl*: cryst. from EtOH. M.p.  $224^\circ$ . Sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>.

Charlesworth, Robinson, *J. Chem. Soc.*, 1933, 269.

### Rochelle Salt.

See under Tartaric Acid.

### Rodinal.

See *p*-Aminophenol.

### Rongalite.

See under Formaldehyde.

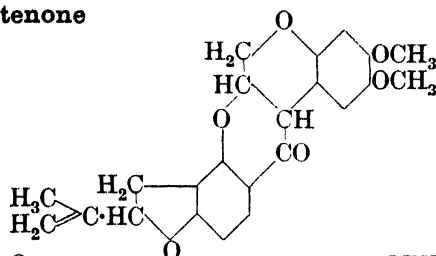
### Rosilic Acid.

9-Hydroxystearic Acid, *q.v.*

### Rotenic Acid.

See Isotubaic Acid.

Rotenone



$C_{23}H_{22}O_6$

MW, 394

Occurs in resin from root of *Derris elliptica*, Benth. Needles or plates from  $Me_2CO.Aq.$  M.p.  $163^\circ$ . B.p.  $210-20^\circ/0.5$  mm. Sol. ord. org. solvents. Sol. in EtOH reduces Fehling's and Tollen's.  $[\alpha]_D^{20} - 225.2^\circ$  in  $C_6H_6$ . Powerful contact insecticide and fish poison.

Oxime: (i) m.p.  $249^\circ$ . (ii) M.p.  $230^\circ$ .

Hydrazone: (i) m.p.  $258^\circ$  decomp. (ii) M.p.  $229^\circ$ .

Phenylhydrazone: (i) m.p.  $255^\circ$  ( $245^\circ$ ). (ii) M.p.  $203^\circ$ .

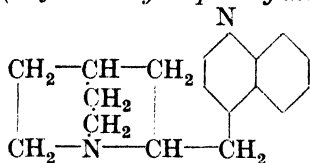
Takei, Miyajima, Ono, *Sci. Papers Inst. Phys. Chem. Research, Tokyo*, 1932, 19, 1, (Review and Bibl.).

La Forge, Haller, Smith, *Chemical Reviews*, 1933, 12, 181 (Review).

Roteol.

See Isotubanol.

Ruban (2-Quinuclidyl-4-quinolylmethane)



$C_{17}H_{20}N_2$

MW, 252

d.-

Oil.  $[\alpha]_D^{16} + 80.5^\circ$ .

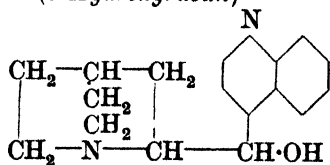
l.-

Oil.  $[\alpha]_D^{16} - 78.4^\circ$ .

Rabe, Riza, *Ann.*, 1932, 496, 151.

Rabe, *Ber.*, 1922, 55, 523, 532.

Rubanol (9-Hydroxyruban)



$C_{17}H_{20}ON_2$

MW, 268

d : d.-

Cryst. from EtOH. M.p.  $229.5-230^\circ$  decomp. Sol. EtOH. Spar. sol.  $Et_2O$ .  $[\alpha]_D^{16} + 132.5^\circ$  in EtOH.

l-Acid tartrate: m.p.  $186-7^\circ$  decomp.  $[\alpha]_D^{15} + 84.7^\circ$  in  $H_2O$ .

d : l.-

Prisms from  $Et_2O$ . M.p.  $118-19^\circ$ . Sol. EtOH,  $Et_2O$ .  $[\alpha]_D^{16} + 14.3^\circ$  in EtOH.

Di-[dibenzyl]-tartrate: m.p.  $189-91^\circ$  decomp.  $[\alpha]_D^{18} - 47.9^\circ$  in MeOH.

l : d.-

M.p.  $117-18^\circ$ .  $[\alpha]_D^{18} - 14.9^\circ$  in EtOH.

Di-[dibenzyl]-tartrate: m.p.  $189-91^\circ$  decomp.  $[\alpha]_D^{23} + 48.0^\circ$  in MeOH.

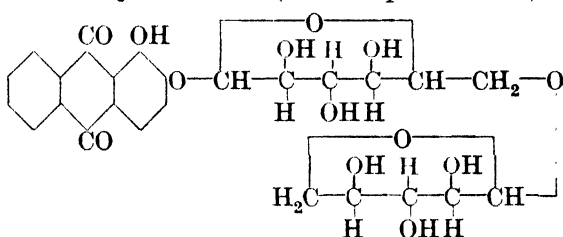
l : l.-

M.p.  $228.5-230.5^\circ$  decomp.  $[\alpha]_D^{15} - 131.8^\circ$  in EtOH.

d-Acid tartrate: m.p.  $186-8^\circ$  decomp.  $[\alpha]_D^{15} - 83.9^\circ$  in  $H_2O$ .

Rabe, Riza, *Ann.*, 1932, 496, 158.

Ruberythric Acid (Alizarin primveroside)



$C_{25}H_{26}O_{13}$

MW, 534

Occurs in *Rubia tinctorum*, Linn. Yellow prisms from  $H_2O$ . M.p.  $258-60^\circ$ . Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ . Insol.  $C_6H_6$ . Sol. alkalis  $\rightarrow$  red col. Hyd.  $\rightarrow$  alizarin + glucose + xylose.

Octa-acetyl deriv.: m.p.  $230^\circ$ .

Penta-Me ether:  $C_{30}H_{38}O_{14}$ ; MW, 622. Pale yellow needles from MeOH. M.p.  $170-80^\circ$ .

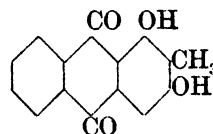
Richter, *J. Chem. Soc.*, 1936, 1701 (Bibl.).

Jones, Robertson, *J. Chem. Soc.*, 1933, 1167.

Zemplén, Müller, *Ber.*, 1929, 62, 2107.

Glaser, Kahler, *Ber.*, 1927, 60, 1349.

Rubiadin (2-Methylpurpuroxanthin, 2-methyl-xanthopurpurin, 1 : 3-dihydroxy-2-methylantraquinone)



$C_{15}H_{10}O_4$

MW, 254

Occurs in root of *Rubia tinctorum*, Linn. (madder). Yellow plates from AcOH. M.p.  $290^\circ$ .

3-Acetyl: m.p.  $191^\circ$ .

Diacetyl: m.p.  $225^\circ$ .

3- $\beta$ -Glucoside: occurs in *Rubia tinctorum*, Linn.  $C_{21}H_{20}O_9$ . MW, 416. Yellow needles from EtOH. M.p. 270–1°. Tetra-acetyl: m.p. 230°.

1:3-Di- $\beta$ -glucoside: m.p. 248°.

Primveroside: occurs in *Galium verum*, Linn.  $C_{26}H_{28}O_{13}$ . MW, 548. Yellow plates. M.p. 248–50°. Sol. hot  $H_2O$ . Hyd.  $\rightarrow$  rubiadin-3-glucoside + xylose.

1-Me ether:  $C_{16}H_{12}O_4$ . MW, 268. Yellow needles from EtOH. M.p. 291°. Acetyl: m.p. 174°.

3-Me ether: yellow plates from AcOH. M.p. 186°. Acetyl: m.p. 200°.

Di-Me ether:  $C_{17}H_{14}O_4$ . MW, 282. Yellow needles from MeOH. M.p. 158°.

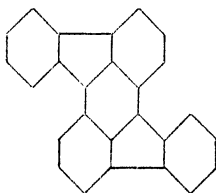
Jones, Robertson, *J. Chem. Soc.*, 1930, 1699.

Kusaka, *Chem. Zentr.*, 1935, II, 3381.

Mitter, Biswas, *J. Indian Chem. Soc.*, 1930, 7, 839.

Hill, Richter, *J. Chem. Soc.*, 1936, 1714.

### Rubicene



$C_{26}H_{14}$

MW, 326

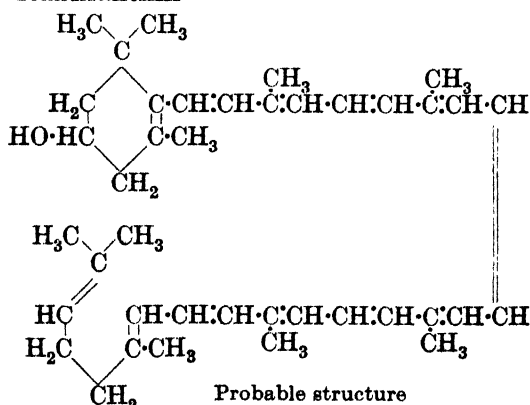
Red needles from  $PhNO_2$ . M.p. 306°. Sol. hot  $PhNO_2$ . Spar. sol.  $C_6H_6$ . Insol. EtOH,  $Et_2O$ , pet. ether. Dil. sols. show intense yellow fluor.

Scholl, Meyer, *Ber.*, 1932, 65, 926.

Eckert, *J. prakt. Chem.*, 1929, 121, 278.

v. Braun, *Ber.*, 1934, 67, 217.

### Rubixanthin



Probable structure

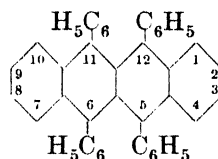
$C_{40}H_{56}O$

MW, 552

Pigment of ripe fruit of *Rosa rubinosa*. Dark red needles from  $C_6H_6$ -MeOH. M.p. 160°. Absorption maxima in  $CS_2$  at 533, 494 and 461 m $\mu$ .

Kuhn, Grundmann, *Ber.*, 1934, 67, 339.

**Rubrene** (5:6:11:12-Tetraphenylnaphthacene)



$C_{42}H_{28}$

MW, 532

Red cryst. M.p. 331°. Sol.  $C_6H_6$ ,  $CS_2$ . Sols. are orange when conc., pink with yellow fluor. when dil.

Koblitz, Witmeyer, *Ber.*, 1936, 69, 1806.

Robin, *Compt. rend.*, 1929, 189, 337.

Allen, Gilman, *J. Am. Chem. Soc.*, 1936, 58, 937.

Bergmann, Herlinger, *J. Chem. Physics*, 1936, 4, 532.

Dufraisse, *Bull. soc. chim.*, 1930, 47, 216.

### Rubreserine

$C_{13}H_{16}O_2N_2$

MW, 232

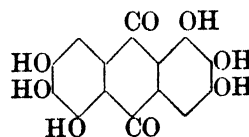
Oxidation product of eseroline. Red needles +  $1H_2O$  from  $H_2O$ . M.p. anhyd. 152°. Sol.  $H_2O$ , EtOH,  $CHCl_3$ , hot  $C_6H_6$ . Insol.  $Et_2O$ , pet. ether.  $B, HCl$ : red cryst. +  $1H_2O$ . M.p. 185° decomp.

$B, H AuCl_4$ : decomp. at 190–5°.

Picrate: decomp. at 198°.

Salway, *J. Chem. Soc.*, 1912, 101, 984.

**Rufigallic Acid** (*Rufigallol*, 1:2:3:5:6:7-hexahydroxyanthraquinone)



$C_{14}H_8O_8$

MW, 304

Red cryst. Sublimes with decomp. on heating. Sol. EtOH,  $Et_2O$ . Sol. alkalis  $\rightarrow$  violet col. Heat of comb.  $\bar{C}_p$  1252.4 Cal.

Tetra-Me ether:  $C_{18}H_{16}O_8$ . MW, 360. Yellow leaflets from AcOEt. M.p. 235–7°. Di-acetyl deriv.: m.p. 262°. Monobenzoyle deriv.: m.p. 190–205°.

Penta-Me ether:  $C_{19}H_{18}O_8$ . MW, 374. Yellow needles from AcOEt. M.p. 192–4°.

**Hexa-Me ether**:  $C_{20}H_{30}O_8$ . MW, 388. Yellow needles. M.p.  $245^\circ$  ( $240^\circ$ ).

**Tri-Et ether**:  $C_{20}H_{20}O_8$ . MW, 388. Orange-red needles from EtOH. M.p.  $195^\circ$ .

**Tetra-Et ether**:  $C_{22}H_{24}O_8$ . MW, 416. Red needles from EtOH. M.p. above  $180^\circ$ . **Di-acetyl deriv.**: m.p.  $230-5^\circ$ .

**Hexa-Et ether**:  $C_{26}H_{32}O_8$ . MW, 472. Orange-yellow needles from EtOH.Aq. M.p. above  $140^\circ$ .

**Hexa-acetyl**: m.p.  $282-3^\circ$ .

Segui, *Chem. Abstracts*, 1934, **28**, 7257.

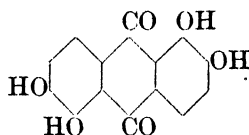
Fischer, Gross, *J. prakt. Chem.*, 1911, **84**, 369.

Schering, D.R.P., 151,724, (*Chem. Zentr.*, 1904, I, 1586).

### Rufigallol.

See Rufigallic Acid.

**Rufiopin** (1 : 2 : 5 : 6-Tetrahydroxyanthraquinone)



$C_{14}H_8O_6$

MW, 272

Orange-red needles from Py. M.p.  $340^\circ$  ( $316-18^\circ$ ). Sol. hot  $H_2O$ . Mod. sol. EtOH, AcOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .

1 : 2 : 6-Tri-Me ether:  $C_{17}H_{14}O_6$ . MW, 314. Orange-red needles from AcOH. M.p.  $245-6^\circ$ .

**Tetra-acetyl**: m.p.  $260-3^\circ$  decomp. ( $238^\circ$ ,  $260-75^\circ$  decomp.).

Heller, *Z. angew. Chem.*, 1929, **42**, 170.

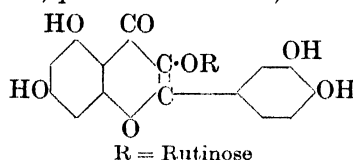
Puntambeker, Adams, *J. Am. Chem. Soc.*, 1927, **49**, 487.

Marshall, *J. Chem. Soc.*, 1937, 254.

### Rufol.

See 1 : 5-Dihydroxyanthracene.

**Rutin** (Eldrin, violaquercitrin, osyritrin, myrticolarin, quercetin rutinoside)



R = Rutinose

$C_{27}H_{30}O_{16}$

MW, 610

Occurs in tomato stems, tobacco leaves, rue leaves and many flowers. Yellow needles +  $2H_2O$  from  $H_2O$ . Softens below  $188^\circ$ , not completely melted at  $190^\circ$ . Sol. hot EtOH. Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ ,  $CS_2$ .  $FeCl_3 \rightarrow$  intense green col.

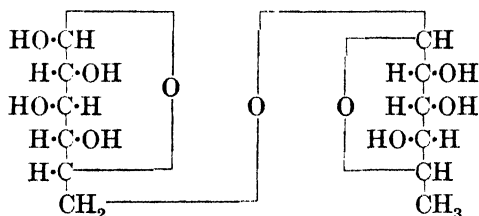
Charaux, *Bull. soc. chim. biol.*, 1924, **6**, 641.

Sanso, Lloyd, *J. Biol. Chem.*, 1924, **58**, 737.

Perkin, *J. Chem. Soc.*, 1900, **97**, 1788.

Zemplén, Gerecs, *Ber.*, 1935, **68**, 1318.

### Rutinose



$C_{12}H_{22}O_{10}$

MW, 326

Hygroscopic powder. Softens at  $140^\circ$ , m.p.  $189-92^\circ$  decomp. Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .  $[\alpha]_D^{20} + 3.24^\circ \rightarrow -0.81^\circ$  in  $H_2O$ ,  $[\alpha]_D^{20} - 10.0^\circ$  in EtOH. Reduces Fehling's.

**Hepta-acetyl deriv.**: m.p.  $168-9^\circ$ .  $[\alpha]_D - 28.84^\circ$  in  $CHCl_3$ . **Me ether**: m.p.  $139.5-140^\circ$ .  $[\alpha]_D - 45.19^\circ$ .

Zemplén, Gerecs, *Ber.*, 1935, **68**, 1319.

Charaux, *Compt. rend.*, 1924, **178**, 1312.

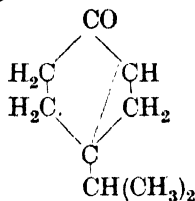
### Rutylidene.

See Nonylacetylene.



## S

## Sabinaketone

 $C_9H_{14}O$ 

MW, 138

l.

Prisms from  $H_2O$ . M.p.  $17^\circ$ . B.p.  $218-19^\circ$ .  $D^{20}_D$  0.9555.  $n^{20}_D$  1.4700. Spar. sol.  $H_2O$ .

Semicarbazone: cryst. from MeOH. M.p.  $141-2^\circ$ .

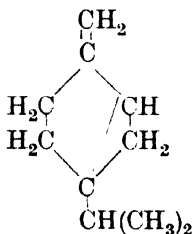
Schmidt, *Z. angew. Chem.*, 1929, **42**, 127.

Wallach, *Ann.*, 1908, **359**, 266.

## Sabinane.

See Thujane.

## Sabinene

 $C_{10}H_{16}$ 

MW, 136

d.

Constituent of various essential oils, e.g. *Juniperus sabina*. B.p.  $163-5^\circ$ ,  $66^\circ/30$  mm.  $D^{20}_D$  0.842.  $n^{20}_D$  1.4678.  $[\alpha]_D^{15} + 80.17^\circ$  ( $89.07^\circ$ ).

l.

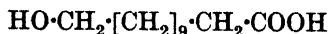
B.p.  $162-6^\circ$ .  $D^{20}_D$  0.8468.  $[\alpha]_D^{15} - 42.5^\circ$ .

Hydrate: methylsabinaketol. Cryst. resembling terpineol in odour. M.p.  $38-9^\circ$ . B.p.  $195-201^\circ$  part. decomp.  $[\alpha]_D^{15} + 53.67^\circ$  in  $Et_2O$ .

Semmler, *Ber.*, 1900, **33**, 1463.

Wallach, *Ann.*, 1907, **357**, 64, 77.

## Sabinic Acid (11-Hydroxylauric acid)

 $C_{12}H_{24}O_3$ 

MW, 216

Cryst. from  $C_6H_6$  or AcOEt, m.p.  $84^\circ$ : needles from MeOH.Aq, m.p.  $78-9^\circ$ . Sol. EtOH.

Me ester:  $C_{13}H_{26}O_3$ . MW, 230. M.p.  $44-5^\circ$  ( $34-5^\circ$ ). B.p.  $160^\circ/7$  mm. ( $164-6^\circ/3$  mm.).

Phenylurethane: m.p.  $64-5^\circ$ .

Anilide: prisms from pet. ether. M.p.  $87^\circ$ .

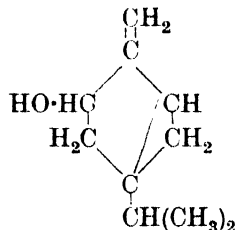
Acetyl: m.p.  $49-49.2^\circ$  ( $43^\circ$ ). B.p.  $202-3^\circ$ .

Chuit, Hausser, *Helv. Chim. Acta*, 1929, **12**, 477.

Bhattacharya, Saletore, Simonsen, *J. Chem. Soc.*, 1928, 2679.

Lycan, Adams, *J. Am. Chem. Soc.*, 1929, **51**, 628.

## Sabinol

 $C_{10}H_{16}O$ 

MW, 152

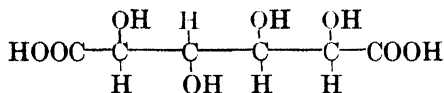
Constituent of oils of *Juniperus sabina* and *Sabina officinalis*. B.p.  $208^\circ$ ,  $77-8^\circ/3$  mm.  $D^{15}_D$  0.9518.  $n^{15}_D$  1.4895.  $[\alpha]_D^{15} + 17.04^\circ$ .

Acetyl: b.p.  $225^\circ$ ,  $81-2^\circ/3$  mm.  $D^{15}_D$  0.972.

Acid phthalate: needles from pet. ether. M.p.  $94-5^\circ$ .  $[\alpha]_D^{15} - 14.63^\circ$ .

Fromm, *Ber.*, 1898, **31**, 2025.

## Saccharic Acid (Tetrahydroxyadipic acid)

 $C_6H_{10}O_8$ 

MW, 210

d.

Needles from 95% EtOH. M.p.  $125-6^\circ$ .  $[\alpha]_D^{19} + 6.86^\circ \rightarrow +20.60^\circ$  in  $H_2O$ . Sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ .  $k$  (first) =  $1.0 \times 10^{-5}$  at  $25^\circ$ .  $HNO_3 \rightarrow d$ -tartaric, racemic, and oxalic acids.  $KMnO_4 \rightarrow d$ -tartaric and oxalic acids.

Cinchonine salt:  $2C_{19}H_{22}ON_2 \cdot C_6H_{10}O_8$ . Cryst. from  $H_2O$ . Decomp. about  $190^\circ$ .  $[\alpha]_D^{20} + 149.1^\circ$  in  $H_2O$ .

Quinine salt:  $2C_{20}H_{24}O_2N_2 \cdot C_6H_{10}O_8$ . Needles. M.p.  $174^\circ$ .

Di-Me ester:  $C_8H_{14}O_8$ . MW, 238. Tetra-Me ether: prisms from  $Et_2O$ . M.p.  $77-8^\circ$  ( $68^\circ$ ). B.p.  $150^\circ/1$  mm.  $[\alpha]_D^{15} + 8.88^\circ \rightarrow +10.26^\circ$  in  $H_2O$ . Sol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ .

Di-Et ester:  $C_{10}H_{18}O_8$ . MW, 266. Cryst. mass. Sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ . Tetra-acetyl: plates from EtOH or prisms from  $Et_2O$ . M.p.  $61^\circ$ . Sol. hot EtOH,  $Et_2O$ . Insol.  $H_2O$ .

**Amide**:  $C_6H_{11}O_7N$ . MW, 209. Needles. M.p.  $135^\circ$  decomp.  $[\alpha]_D^{19} + 22.5^\circ$  in  $H_2O$ . Sol. hot  $H_2O$ . Spar. sol. EtOH.

**Diamide**:  $C_6H_{12}O_6N_2$ . MW, 208. Cryst. from EtOH. M.p.  $172-3^\circ$ .  $[\alpha]_D^{20} + 13.3^\circ$  in  $H_2O$ . **Tetra-Me ether**: plates from  $H_2O$ . M.p.  $237-9^\circ$ .  $[\alpha]_D^{18} + 12.22^\circ$  in  $H_2O$ .

**$\gamma$ -Lactone**:  $C_6H_8O_7$ . MW, 192. Cryst. M.p.  $130-2^\circ$ . Sol.  $H_2O$ .  $[\alpha]_D^{25} + 40.8^\circ \rightarrow + 22.7^\circ$  in  $H_2O$ .

**Diphenylhydrazide**: yellow plates. Decomp. at  $210^\circ$ . Insol.  $H_2O$ , EtOH, Et<sub>2</sub>O.

l.

Free acid not isolated.

**KH salt**: needles or prisms from hot  $H_2O$ . Mod. sol.  $H_2O$ .

**Diphenylhydrazide**: yellow plates. M.p.  $213-14^\circ$  decomp.

dl.

Syrup.

**KH salt**: needles. Mod. sol.  $H_2O$ .

**Diphenylhydrazide**: laminæ. M.p.  $209-10^\circ$  decomp.

Sohst, Tollens, *Ann.*, 1888, **245**, 1.

Henneberg, Tollens, *Ann.*, 1896, **292**, 40.

Fischer, *Ber.*, 1890, **23**, 2621.

Fischer, Stahel, *Ber.*, 1891, **24**, 534.

Kiliani, *Ber.*, 1925, **58**, 2344.

Rehorst, *Ber.*, 1928, **61**, 163.

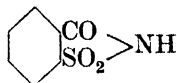
Haworth, Loach, Long, *J. Chem. Soc.*, 1927, 3154.

Karrer, Peyer, *Helv. Chim. Acta*, 1922, **5**, 577.

Bergmann, *Ber.*, 1921, **54**, 1380, 2653.

Hudson, Komatsu, *J. Am. Chem. Soc.*, 1919, **41**, 1147.

**Saccharin** (*Benzoic sulphimide, o-sulphobenzoic imide*)



$C_7H_5O_3NS$

MW, 183

Prisms from EtOH, leaflets from  $H_2O$ . M.p.  $224^\circ$  decomp. Sol. 250 parts cold  $H_2O$ , 40 parts cold EtOH, 221 parts hot  $C_6H_6$ . Sublimes in vacuo. Forms salts with  $NH_3$  and org. bases. All salts intensely sweet. Hot alkalis or hot conc. HCl  $\rightarrow$  o-sulphobenzoic acid.

**Oxime**: m.p.  $208-10^\circ$  decomp. **Acetate**: m.p.  $225^\circ$  decomp. **Benzoate**: m.p.  $250^\circ$  decomp.

**N-Me**:  $C_8H_7O_3NS$ . MW, 197. Needles from  $H_2O$ . M.p.  $131-2^\circ$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold  $H_2O$ .

Dict. of Org. Comp.—III.

**N-Et**:  $C_9H_9O_3NS$ . MW, 211. Needles from  $H_2O$ . M.p.  $93-4^\circ$ . Very sol. EtOH, Et<sub>2</sub>O. Mod. sol. hot  $H_2O$ .

Zaikov, Sokolov, *Chem. Abstracts*, 1928, **22**, 2933.

Brackett, Hayes, *Am. Chem. J.*, 1887, **9**, 405.

Zil'berg, *Chem. Abstracts*, 1935, **29**, 1794.

Roost, *Chem. Abstracts*, 1928, **22**, 4114.

Orelup, U.S.P. 1,601,505, (*Chem. Zentr.*, 1927, II, 2115).

### Saccharose.

See Sucrose.

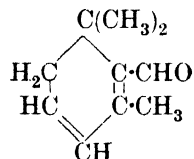
### S-Acid.

See 1-Amino-8-naphthol-4-sulphonic Acid.

### 2S-Acid.

See 1-Amino-8-naphthol-2:4-disulphonic Acid.

### Safranöl



$C_{10}H_{14}O$

MW, 150

Hydrolysis product of picrocrocin. B.p.  $70^\circ/1$  mm.  $D_4^{19}$  0.9734.  $n_D^{19}$  1.5281.

**Oxime**: prisms from pet. ether. M.p.  $65^\circ$ .

**Semicarbazone**: cryst. from MeOH. M.p.  $175^\circ$ .

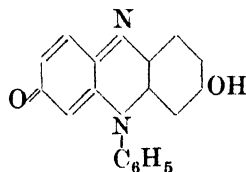
**Thiosemicarbazone**: cryst. from  $C_6H_6$ . M.p.  $199-200^\circ$ .

**2:4-Dinitrophenylhydrazone**: red cryst. from MeOH. M.p.  $186^\circ$ .

Kuhn, Wendt, *Ber.*, 1936, **69**, 1549.

Kuhn, Winterstein, *Ber.*, 1934, **67**, 354.

### Safranöl



$C_{18}H_{12}O_2N_2$

MW, 288

Needles. Does not melt below  $330^\circ$ . Very spar. sol. Et<sub>2</sub>O. Insol.  $H_2O$ , EtOH, AcOH. Sol. alkalis.

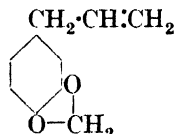
**Me ether**:  $C_{19}H_{14}O_2N_2$ . MW, 302. M.p.  $266^\circ$ .

**Et ether**:  $C_{20}H_{16}O_2N_2$ . MW, 316. Bronze prisms from EtOH. M.p.  $265^\circ$  decomp.

*Acetyl*: red prisms with greenish lustre. M.p. 265–8°.

Jaubert, *Ber.*, 1895, **28**, 273.

**Safrol** (3 : 4-Methylenedioxyallylbenzene)



$C_{10}H_{10}O_2$

MW, 162

Constituent of several essential oils, e.g. *Sassafras officinalis*. Monoclinic prisms. Setting-point 11.2°. B.p. 231.5–232°, 104–5°/6 mm.  $D_4^{20}$  1.100.  $n_D^{20}$  1.5383. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Picrate*: red cryst. from EtOH. M.p. 75°.

Perkin, Trikojus, *J. Chem. Soc.*, 1927, 1663.

**Saiodin**.

See under Iodobehenic Acid.

**Sakuranin** (Sakuranetin-5-glucoside)

$C_{22}H_{24}O_{10}$

MW, 448

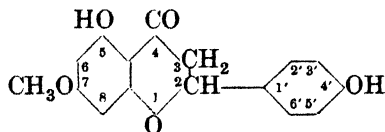
Constituent of rind of *Prunus yeodensis*. Needles from EtOH or AcOEt. M.p. 212°. Very sol. 60% EtOH, Py. Less sol. EtOH. Insol. H<sub>2</sub>O, Et<sub>2</sub>O.  $[\alpha]_D^{25}$  –106.6° in Me<sub>2</sub>CO. Alkalis → intense yellow sols. Conc. H<sub>2</sub>SO<sub>4</sub> → intense brown col. → yellow on adding more acid. Alc. FeCl<sub>3</sub> → yellow col.

*Oxime*: needles from EtOH. M.p. 110°. Conc. HNO<sub>3</sub> → green col.

Asahina, *Chem. Zentr.*, 1908, II, 253.

Asahina, Shinoda, Inubuse, *Chem. Zentr.*, 1928, I, 1672.

**Sakuranetin** (Naringenin 7-methyl ether, 5 : 4'-dihydroxy-7-methoxyflavanone)



$C_{18}H_{14}O_5$

MW, 286

Needles from EtOH.Aq., dry Et<sub>2</sub>O, or C<sub>6</sub>H<sub>6</sub>. M.p. 150°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, AcOEt, Py. Spar. sol. boiling H<sub>2</sub>O. Fuming HNO<sub>3</sub> → deep indigo-blue col. → violet on standing. Alkalis → intense yellow sols. decomp. by CO<sub>2</sub> and weak acids. Mg + alc. HCl → purplish-red col. KOH fusion → phloroglucinol + *p*-hydroxybenzoic acid. Tasteless.

4'-Me ether: see under Isosakuranetin.

Di-Me ether: see under Isosakuranetin.

*Oxime*: needles + 2H<sub>2</sub>O from EtOH.Aq., m.p. 120°: needles from AcOH, m.p. 195–6° decomp.

5-Glucoside: see Sakuranin.

Diacetyl: m.p. 97°.

Shinoda, Sato, *Chem. Zentr.*, 1929, I, 244.

See also last reference above.

**Salacetol** (Acetol salicylate, hydroxyacetone salicylate, β-ketopropyl salicylate)

COO·CH<sub>2</sub>·CO·CH<sub>3</sub>



$C_{10}H_{10}O_4$

MW, 194

Needles from EtOH. M.p. 71°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Spar. sol. ligroin. Very spar. sol. hot H<sub>2</sub>O. Triboluminescent.

Fritsch, D.R.P., 70,054.

**Salazine** (Salicylaldazine, 2 : 2'-dihydroxybenzaldazine)



$C_{14}H_{12}O_2N_2$

MW, 240

Yellow needles or leaflets from EtOH. M.p. 214°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, alkalis. Mod. sol. EtOH. Insol. H<sub>2</sub>O. Sublimes.

Di-Me ether: C<sub>16</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>. MW, 268. Yellow needles from EtOH. M.p. 178° (141°).

Di-Et ether: C<sub>18</sub>H<sub>20</sub>O<sub>2</sub>N<sub>2</sub>. MW, 296. Yellow cryst. from EtOH. M.p. 136° (130°).

Dibenzyl ether: yellow plates from C<sub>6</sub>H<sub>6</sub>. M.p. 158°.

Diacetyl: plates from CHCl<sub>3</sub>. M.p. 191°.

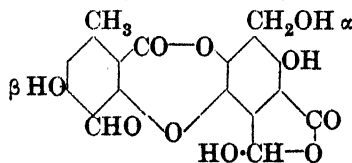
Dibenzoyl: yellow needles from C<sub>6</sub>H<sub>6</sub>. M.p. 188–9°.

Widman, *Ber.*, 1919, **52**, 1658.

Borsche, *Ber.*, 1901, **34**, 4299; 1921, **54**, 668.

Curtius, Jay, *J. prakt. Chem.*, 1889, **39**, 48.

**Salazinic Acid** (Saxatilisic acid)



Probable structure

$C_{18}H_{12}O_{10}$

MW, 388

Isolated from *Parmelia conspersa*, Ach. Needles or plates from  $\text{Me}_2\text{CO}$ . M.p.  $270^\circ$  (decomp. at  $260^\circ$ ). Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ .

$\alpha$ -Me ether:  $\text{C}_{19}\text{H}_{14}\text{O}_{10}$ . MW, 402. Needles from 80%  $\text{Me}_2\text{CO}$ . M.p.  $210^\circ$  decomp. Sol.  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{AcOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{C}_6\text{H}_6$ , pet. ether. *Dianil*: yellow prisms from  $\text{Me}_2\text{CO}$ . M.p.  $169^\circ$  decomp.

$\beta$ -Me ether: needles from 80%  $\text{Me}_2\text{CO}$ . M.p.  $250-2^\circ$  decomp. Sol.  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{AcOH}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether. *Penta-acetyl*: plates from  $\text{Me}_2\text{CO}$ . M.p.  $228^\circ$  decomp. *Dianil*: yellow prisms from  $\text{Me}_2\text{CO}$ . M.p.  $231^\circ$  decomp.

$\alpha$ -Acetyl: needles from  $\text{AcOH}$ . M.p.  $275-6^\circ$  decomp. Sol.  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ .

*Tetra-acetyl*: m.p.  $205-6^\circ$ .

*Hexa-acetyl*: needles from  $\text{Me}_2\text{CO}$ . M.p.  $178^\circ$ .

*Di-phenylhydrazone*: yellow prisms. De-comp. at  $295^\circ$ .

*Dianil*: decomp. at  $280^\circ$ .

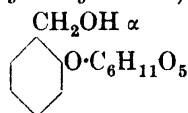
Asahina, Tukamoto, *Ber.*, 1934, **67**, 965.

Koller, Kutzelnigg, *Monatsh.*, 1934, **65**, 92.

Asahina, Asano, *Ber.*, 1933, **66**, 895.

Fuzikawa, Ishiguro, *Chem. Zentr.*, 1937, I, 2996.

### Salicin (*Saligenin glucoside*)



$\text{C}_{13}\text{H}_{18}\text{O}_7$  MW, 286

Glucoside of poplar and willow bark. Needles from  $\text{H}_2\text{O}$ . M.p.  $201^\circ$  ( $198^\circ$ ). Sol. 24 parts  $\text{H}_2\text{O}$  at  $25^\circ$ .  $[\alpha]_D^{20} - 62.56^\circ$  in  $\text{H}_2\text{O}$ . Hydrolysis by emulsin  $\rightarrow$  glucose + saligenin.  $\text{H}_2\text{SO}_4 \rightarrow$  purple col. Possesses burning taste.

*Penta-Me ether*:  $\text{C}_{18}\text{H}_{28}\text{O}_7$ . MW, 356. Needles from pet. ether. M.p.  $62-4^\circ$ .  $[\alpha]_D - 52.1^\circ$  in  $\text{MeOH}$ .

*Tetra-acetyl*: needles from  $\text{H}_2\text{O}$ . M.p.  $130^\circ$  ( $126^\circ$ ). Insol. pet. ether.  $\alpha$ -Me ether: plates from  $\text{EtOH}$ . M.p.  $142^\circ$ .  $\alpha$ -Allyl ether: needles from  $\text{EtOH}$ . M.p.  $139.5^\circ$ .  $\alpha$ -Benzyl ether: plates from  $\text{EtOH}$  or pet. ether. M.p.  $94.5-95^\circ$ .  $\alpha$ -Phenyl ether: yellow needles from  $\text{EtOH}$ . M.p.  $161^\circ$ .

*Penta-acetyl*: cryst. from  $\text{EtOH}$ . M.p.  $130^\circ$ .  $[\alpha]_D^{25} - 18.5^\circ$  in  $\text{CHCl}_3$ .

*Benzoyl*: see Populin.

Kunz, *J. Am. Chem. Soc.*, 1926, **48**, 262.

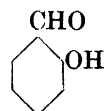
Zemplén, Braun, *Ber.*, 1925, **58**, 1406.

Irvine, Rose, *J. Chem. Soc.*, 1906, **89**, 814.

### Salicyl Alcohol.

See Saligenin.

### Salicylaldehyde (*o*-Hydroxybenzaldehyde)



$\text{C}_7\text{H}_6\text{O}_2$

MW, 122

Oil. F.p.  $-7^\circ$ . B.p.  $197^\circ$ ,  $93^\circ/25$  mm.,  $86^\circ/18$  mm.  $D_4^{20}$  1.1674.  $n_D^{20}$  1.574. Heat of comb.  $C_p$  807.3 Cal. Volatile in steam. Does not reduce Fehling's.  $\text{H}_2\text{O}_2 \rightarrow$  2:3-dihydroxybenzaldehyde.  $\text{KOH} \rightarrow$  salicylic acid.  $\text{NaHg} \rightarrow$  saligenin.  $\text{FeCl}_3 \rightarrow$  violet pol.  $\text{H}_2\text{SO}_4 \rightarrow$  orange col.

*Me ether*: see *o*-Methoxybenzaldehyde.

*Et ether*: *o*-ethoxybenzaldehyde.  $\text{C}_9\text{H}_{10}\text{O}_2$ . MW, 150. M.p.  $20-2^\circ$ . B.p.  $247-9^\circ$ ,  $143-7^\circ/25$  mm. *Diacetate*: prisms. M.p.  $88-9^\circ$ .

*Oxime*: prisms from pet. ether. M.p.  $57-9^\circ$ .

*Semicarbazone*: needles from  $\text{EtOH}$ . M.p.  $219^\circ$ .

*Allyl ether*:  $\text{C}_{10}\text{H}_{10}\text{O}_2$ . MW, 162. B.p.  $130^\circ/10$  mm.

*Benzyl ether*:  $\text{C}_{14}\text{H}_{12}\text{O}_2$ . MW, 212. Prisms from  $\text{EtOH}$ . M.p.  $46^\circ$ . *Oxime*: plates from pet. ether. M.p.  $71.5^\circ$ .

*Acetyl*: needles from pet. ether. M.p.  $38-9^\circ$ . B.p.  $142^\circ/18$  mm. *Semicarbazone*: powder from  $\text{Me}_2\text{CO}$ . M.p.  $167^\circ$ .

*Triacetate*: *o*-acetoxybenzylidene diacetate. Prisms from  $\text{EtOH}$ . M.p.  $107^\circ$ .

*Benzoyl*: b.p. above  $300^\circ$ .

*p*-Nitrobenzoyl: yellow plates. M.p.  $128^\circ$ .

*Oxime*: prisms from  $\text{C}_6\text{H}_6$ -pet. ether. M.p.  $63^\circ$  ( $57^\circ$ ). *Me ether*: ( $>\text{N}\cdot\text{OCH}_3$ ). Prisms. M.p.  $28^\circ$ . B.p.  $107^\circ/14$  mm. *Acetate*: ( $>\text{N}\cdot\text{OCO}\cdot\text{CH}_3$ ). Needles from pet. ether. M.p.  $75^\circ$  ( $69^\circ$ ).

*Semioxamazone*: needles from  $\text{EtOH}$ . Aq. M.p.  $255^\circ$ .

*Semicarbazone*: needles from  $\text{EtOH}$ . M.p.  $230^\circ$ .

*Phenylsemicarbazone*: needles. M.p.  $198-200^\circ$ .

*o*-Tolylsemicarbazone: needles. M.p.  $204.5^\circ$ .

*p*-Tolylsemicarbazone: needles. M.p.  $238-9^\circ$ .

*Thiosemicarbazone*: m.p.  $231^\circ$  (sinters at  $215^\circ$ ).

*Phenylthiosemicarbazone*: needles or plates. M.p.  $183^\circ$ .

*Hydrazone*: plates from  $\text{EtOH}$ . M.p.  $96^\circ$ .

*Phenylhydrazone*: needles or plates. M.p.  $142-3^\circ$ . Sensitive to light. *Dibenzoyl*: m.p.  $170-1^\circ$ .

2 : 4 : 6-Trichlorophenylhydrazone : needles from EtOH or AcOH. M.p. 98.5–99.5°.

o-Bromophenylhydrazone : yellow cryst. M.p. 111–12°.

p-Bromophenylhydrazone : yellow plates. M.p. 175.5°.

2 : 4 : 6-Tribromophenylhydrazone : needles from EtOH. M.p. 100°.

o-Nitrophenylhydrazone : m.p. 193°.

m-Nitrophenylhydrazone : m.p. 197°.

p-Nitrophenylhydrazone : reddish-brown prisms. M.p. 227°.

2 : 4-Dinitrophenylhydrazone : purplish-red needles from EtOH. M.p. 248°.

o-Tolylhydrazone : m.p. 111–12°.

1-Naphthylhydrazone : yellow plates. M.p. 134°.

2-Naphthylhydrazone : yellow needles. M.p. 187°.

Benzoylhydrazone : needles from EtOH. Aq. M.p. 182°.

1-Naphthoylhydrazone : needles. M.p. 235°.

Phenylurethane : m.p. 133°.

p-Toluenesulphonyl : cryst. from MeOH. M.p. 63–4°.

Glucoside : see Helicin.

Azine : see Salazine.

Anil : see Salicylideneaniline.

Claisen, Eisleb, *Ann.*, 1913, **401**, 95.

Brady, Dunn, *J. Chem. Soc.*, 1914, **105**, 825.

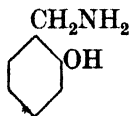
Kawada, Yosida, *Chem. Abstracts*, 1930, **24**, 1040.

Copisarow, *J. Chem. Soc.*, 1929, 588.

B.D.C., E.P., 232,392, (*Chem. Zentr.*, 1925, II, 1226).

Weiss, Donns, U.S.P., 1,380,277, (*Chem. Zentr.*, 1921, IV, 587).

**Salicylamine** (o-Hydroxybenzylamine, ω-amino-o-cresol)



$C_7H_9ON$

MW, 123

Cryst. from EtOH–pet. ether. M.p. 129° after softening at 126°. Sol.  $H_2O$ , EtOH,  $Et_2O$ . Insol. pet. ether. Sublimes readily.  $FeCl_3 \rightarrow$  violet-blue col.  $\rightarrow$  red on heating.

*Me ether* : o-methoxybenzylamine.  $C_8H_{11}ON$ . MW, 137. Liq. B.p. 224°/724 mm. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ . *B.HCl* : prisms from EtOH. M.p. 150°.  $B_2H_2PtCl_6$  : yellow plates +  $2H_2O$  from  $H_2O$ . M.p. 187°.

*Et ether* :  $C_9H_{13}ON$ . MW, 151. Oil.  $B_2H_2PtCl_6$  : yellow cryst. M.p. 182°.

*N-Acetyl* : o-hydroxybenzylacetamide. Plates from  $CHCl_3$ –pet. ether. M.p. 140°.

*Diacetyl deriv.* : plates from  $CHCl_3$ –pet. ether. M.p. 102–3°.

*Benzoyl* : o-hydroxybenzylbenzamide. Plates from  $CHCl_3$ –pet. ether. M.p. 142° (softens at 140°).

*Dibenzoyl deriv.* : needles from EtOH–pet. ether. M.p. 142–3° (softens at 137°).

*B.HCl* : needles from EtOH. Very sol.  $H_2O$ .

*B.HI* : m.p. 184°.

$B_2H_2PtCl_6$  : yellow needles +  $2H_2O$  from  $H_2O$ . M.p. 197° decomp.

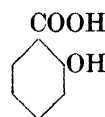
Raiford, Clark, *J. Am. Chem. Soc.*, 1923, **45**, 1740.

Löw, *Monatsh.*, 1891, **12**, 397.

Tiemann, *Ber.*, 1890, **23**, 3017.

Goldschmidt, Ernst, *Ber.*, 1890, **23**, 2744.

**Salicylic Acid** (o-Hydroxybenzoic acid)



$C_7H_6O_3$

MW, 138

Occurs in form of esters in many essential oils and plant products, e.g. oil of wintergreen. Needles from  $H_2O$ . M.p. 159°. B.p. 211°/20 mm. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ . Mod. sol.  $H_2O$ ,  $C_6H_6$ . Volatile in steam. Sublimes in vacuo. Heat of comb.  $C_p$  728.2 Cal.  $k = 1.06 \times 10^{-3}$  at 25°. Above 200°  $\rightarrow$  phenol +  $CO_2$  + a little salol.  $FeCl_3 \rightarrow$  violet col. in  $H_2O$ . Strong antiseptic.

*Me ester* : see Methyl salicylate.

*Et ester* : see Ethyl salicylate.

*Propyl ester* :  $C_{10}H_{12}O_3$ . MW, 180. B.p. 238–40°.  $D^{20}_D$  1.021.

*Isopropyl ester* : b.p. 120–2°/18 mm.  $D^{25}_D$  1.0101.  $n^{25}_D$  1.5003.

*Butyl ester* :  $C_{11}H_{14}O_3$ . MW, 194. M.p. 5–9°. B.p. 259–60°.

*Isoamyl ester* : see Isoamyl salicylate.

*Allyl ester* :  $C_{10}H_{10}O_3$ . MW, 178. B.p. 247–50°, 105°/5 mm.  $D^{15}_D$  1.100.

*Benzyl ester* :  $C_{14}H_{12}O_3$ . MW, 228. B.p. 208°/26 mm., 186°/10 mm., 170–5°/7 mm.  $D^{20}_D$  1.1799.  $n^{20}_D$  1.5805.

2 : 4-Dinitrobenzyl ester : m.p. 168°.

*Phenyl ester* : see Salol.

o-Tolyl ester :  $C_{14}H_{12}O_3$ . MW, 228. M.p. 35°.

m-Tolyl ester : m.p. 74°.

p-Tolyl ester : m.p. 39°.

Salicyl ester : see Diplosal.

Phenacyl ester : m.p. 110°.

1-Naphthyl ester : see under 1-Naphthol.

2-Naphthyl ester : see under 2-Naphthol.

Glycerol ester : trisalicylin. Needles. M.p. 79°.

Chloride : o-hydroxybenzoyl chloride.  $C_7H_5O_2Cl$ . MW, 156.5. Needles. M.p. 19–19.5°. B.p. 92°/15 mm.

Amide :  $C_7H_7O_2N$ . MW, 137. Yellow cryst. M.p. 133°. O-Benzoyl : m.p. 144°. N-Benzoyl : m.p. 208°.

Nitrile : o-cyanophenol.  $C_7H_5ON$ . MW, 119. Prisms from  $C_6H_6$ -pet. ether. M.p. 98°.  $D_4^{20}$  1.1052.  $n_D^{20}$  1.53716. Benzoyl : needles from pet. ether. M.p. 106°.

Anilide : prisms from  $H_2O$ . M.p. 135°.

Piperidide : plates from EtOH. M.p. 142°.

Me ether : see o-Methoxybenzoic Acid.

Et ether : see o-Ethoxybenzoic Acid.

Propyl ether :  $C_{10}H_{12}O_3$ . MW, 180. Plates. M.p. 30°.

Benzyl ether :  $C_{14}H_{12}O_3$ . MW, 228. M.p. 76–7°.

Phenyl ether : see o-Phenoxybenzoic Acid.

Acetyl : see Acetylsalicylic Acid.

Benzoyl : needles. M.p. 132°.

p-Nitrobenzoyl : yellow cryst. from MeOH. M.p. 205°.

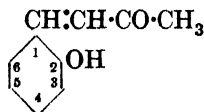
Schott, *Chem. Zentr.*, 1912, I, 754.

Ma, Hoo, Sah, *Chem. Abstracts*, 1934, **28**, 133.

Rosenmund, Harms, *Ber.*, 1920, **53**, 2230.

Pomilio, E.P. 103,739, (*Chem. Abstracts*, 1917, **11**, 1794).

**Salicylideneacetone** (o-Hydroxybenzylideneacetone, methyl 2-hydroxystyryl ketone)



$C_{10}H_{10}O_2$  MW, 162

Needles from ligroin. M.p. 140°. Sol.  $Et_2O$ . Spar. sol. cold  $H_2O$ .

Me ether : o-methoxybenzylideneacetone.  $C_{11}H_{12}O_2$ . MW, 176. Prisms. M.p. 48–50°. B.p. 180–2°/20 mm.  $D_4^{21}$  1.0638.  $n_D^{21}$  1.586. Sol. usual org. solvents.

Oxime : cryst. from  $C_6H_6$ -ligroin. M.p. 84–5°. Sol.  $Et_2O$ . Spar. sol. EtOH,  $C_6H_6$ , ligroin.

Semicarbazone : needles from EtOH. M.p. 206–7° decomp.

Tiemann, Kees, *Ber.*, 1885, **18**, 1966.

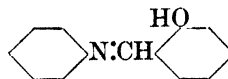
Harries, *Ber.*, 1891, **24**, 3180.

Auwers, *Ann.*, 1917, **413**, 279.

$\omega$ -Salicylideneacetophenone.

See 2-Hydroxychalkone.

**Salicylideneaniline** (o-Hydroxybenzylideneaniline, salicylaldehyde anil)



$C_{13}H_{11}ON$  MW, 197

Yellow plates from MeOH. M.p. 51°. Sol. EtOH. Insol.  $H_2O$ .

B.HCl : yellow needles. M.p. 91°.

Me ether : o-methoxybenzylideneaniline.

$C_{14}H_{13}ON$ . MW, 211. Needles from EtOH. M.p. 44°. B.p. 330–4°, 235–6°/30 mm.

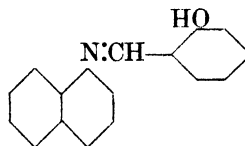
Picrate : yellow needles. M.p. 153–4°.

Schischkow, *Ann.*, 1857, **104**, 373.

Schiff, *Ann.*, 1869, **150**, 194.

Noelting, *Ann. chim.*, 1910, **19**, 540.

**Salicylidene-1-naphthylamine** (o-Hydroxybenzylidene-1-naphthylamine)



$C_{17}H_{13}ON$  MW, 247

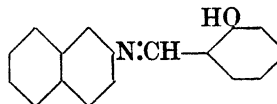
Needles from  $C_6H_6$ . M.p. 53° (45–5°). Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

B.HCl : m.p. 210° decomp.

Pope, Fleming, *J. Chem. Soc.*, 1908, **93**, 1916.

Senier, Shephard, *J. Chem. Soc.*, 1909, **95**, 443.

**Salicylidene-2-naphthylamine** (o-Hydroxybenzylidene-2-naphthylamine)



$C_{17}H_{13}ON$  MW, 247

Needles and prisms from EtOH. M.p. 126°. Sol. EtOH,  $C_6H_6$ ,  $CHCl_3$ , ligroin. Spar. sol.  $Et_2O$ .

Senier, Shephard, *J. Chem. Soc.*, 1909, **95**, 1950.

Emmerich, *Ann.*, 1887, **241**, 351.

**1-Salicylidenepropionic Acid.**

See  $\alpha$ -Methyl-o-coumaric Acid and  $\alpha$ -Methyl-o-coumarinic Acid.

**o-Salicyloylbenzoic Acid.**

See 2'-Hydroxybenzophenone-2-carboxylic Acid.

**Salicyloylglycine.**

See 2-Hydroxyhippuric Acid.

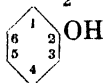
**Salicyloylsalicylic Acid.**

See Diplosal.

 **$\beta$ -Salicyloylstyrene.**

See 2'-Hydroxystyrene.

**Saligenin** (Salicyl alcohol, o-hydroxybenzyl alcohol,  $\omega$ -hydroxy-o-cresol)



$\text{C}_7\text{H}_8\text{O}_2$

MW, 124

Needles or plates from  $\text{H}_2\text{O}$  or  $\text{Et}_2\text{O}$ . M.p.  $87^\circ$ .  $D^{25}_D$  1.1613. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Sublimes in plates.  $\text{H}_2\text{SO}_4 \rightarrow$  red col.  $\text{FeCl}_3 \rightarrow$  blue col. Antiseptic.

$\alpha$ -Me ether:  $\text{C}_8\text{H}_{10}\text{O}_2$ . MW, 138. B.p.  $128-30^\circ/40$  mm.

2-Me ether: o-methoxybenzyl alcohol. B.p.  $248-50^\circ$ ,  $119^\circ/8$  mm.  $D^{17}_D$  1.128.  $n^{17}_D$  1.549. Acetyl: b.p.  $130^\circ/12$  mm.  $D^{17}_D$  1.117.  $n^{17}_D$  1.515. Benzoyl: m.p.  $59^\circ$ .

Di-Me ether:  $\text{C}_9\text{H}_{12}\text{O}_2$ . MW, 152. B.p.  $229-30^\circ$ .

$\alpha$ -Et ether:  $\text{C}_9\text{H}_{12}\text{O}_2$ . MW, 152. B.p.  $111-13^\circ/20$  mm.

2-Et ether: b.p.  $265^\circ$ .

2-Allyl ether:  $\text{C}_{10}\text{H}_{12}\text{O}_2$ . MW, 164. B.p.  $133-50^\circ/9$  mm.

2-Benzoyl: needles from  $\text{C}_6\text{H}_6$ -pet. ether. M.p.  $66^\circ$ .

Carothers, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1680.

Rutovskii, Karolev, *Chem. Abstracts*, 1930, **24**, 24.

Manasse, *Ber.*, 1894, **27**, 2411.

Vavon, *Compt. rend.*, 1912, **154**, 359.

Paal, Senninger, *Ber.*, 1894, **27**, 1084.

**Salinigrin.**

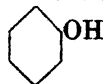
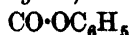
See Piceoside.

**Salipurool.**

See Naringenin.

**Salipyrine.**

See under Antipyrine.

**Salol** (Phenyl salicylate)

$\text{C}_{13}\text{H}_{10}\text{O}_3$

MW, 214

Plates from  $\text{MeOH}$ . M.p.  $43^\circ$  ( $41.7^\circ$ ). B.p.  $173^\circ/12$  mm.  $D^{20}_D$  1.1553. Sol.  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ . Antiseptic.

Me ether:  $\text{C}_{14}\text{H}_{12}\text{O}_3$ . MW, 228. Prisms from  $\text{EtOH}$ . M.p.  $59^\circ$ .

Phenyl ether: see under o-Phenoxybenzoic Acid.

p-Nitrobenzyl ether: cryst. from  $\text{EtOH}$ . Aq. M.p.  $87^\circ$ .

Chloroformyl: m.p.  $90-1^\circ$ .

Acetyl: cryst. from  $\text{EtOH}$ . M.p.  $98^\circ$ . B.p.  $197-8^\circ/11$  mm.

Benzoyl: cryst. from  $\text{EtOH}$ . M.p.  $80.5-81^\circ$ .

Phosphate:  $(\text{HO})_2\text{PO}\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{OC}_6\text{H}_5$ . Cryst. M.p.  $88^\circ$ .

Phosphate, hydrate:  $(\text{HO})_4\text{P}\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{OC}_6\text{H}_5$ . Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $62^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Sol.  $\text{CHCl}_3$ ,  $\text{AcOH}$ , hot  $\text{C}_6\text{H}_6$ .

Phosphate diethyl ester:

$(\text{C}_2\text{H}_5\text{O})_2\text{OP}\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{OC}_6\text{H}_5$ . Oil. B.p.  $105-13^\circ/13$  mm.

Phosphate diphenyl ester:

$(\text{C}_6\text{H}_5\text{O})_2\text{OP}\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{OC}_6\text{H}_5$ . Cryst. from  $\text{EtOH}$ . M.p.  $76-7^\circ$ .

Seifert, *J. prakt. Chem.*, 1885, **31**, 472.

Graebe, Eichengrün, *Ann.*, 1892, **269**, 324.

Chem. Fabr. v. Heyden, D.R.P., 38,973.

Bayer, D.R.P., 206,055, (*Chem. Zentr.*, 1909, I, 703).

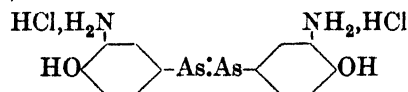
**Salophene.**

See p-Acetylaminophenyl salicylate.

**Saloquinine.**

See under Quinine.

**Salvarsan** (Arsphenamine, "606," dihydrochloride of 3:3'-diamino-4:4'-dihydroxyarseno benzene)



$\text{C}_{12}\text{H}_{14}\text{O}_2\text{N}_2\text{Cl}_2\text{As}_2$

MW, 439

Greyish powder. Decomp. (indefinite) at  $180-95^\circ$ . Sol. hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{MeOH}$ ,  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Used in treatment of protozoal diseases.

Kirchoff, Korsina, Sirkin, D.R.P., 592,870, (*Chem. Zentr.*, 1934, I, 2791).

Christiansen, *J. Am. Chem. Soc.*, 1923, **45**, 1807.

Fargher, Pyman, *J. Chem. Soc.*, 1920, **117**, 376.

**Salvianin chloride.**

See Monardaquin chloride.

## Samandarin

 $C_{19}H_{31}O_2N$ 

MW, 305

Secretion of *Salamander maculosa*, *Salamander atra*, etc. Colourless cryst. from EtOH.Aq.,  $Me_2CO$ , and with 1MeOH from MeOH. M.p. 187–8°. Strongly basic.  $[\alpha]_D^{17} + 43.7^\circ$ . HCl  $\rightarrow$  blue col.

*B,HCl*: m.p. 321–2°.

*N-Me*:  $C_{20}H_{33}O_2N$ . MW, 319. *B,HCl*: cryst. from EtOH.Aq. M.p. 300–2° decomp. *Methiodide*: cryst. from  $H_2O$ . M.p. 271–2°.

*N-Nitroso*: cryst. from EtOH.Aq. M.p. 164–5°.

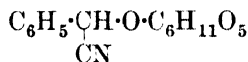
*Monoformyl deriv.*: cryst. from MeOH.Aq. M.p. 148–50°.

*Diformyl deriv.*: needles from MeOH. M.p. 256–8°.

*Diacetyl deriv.*: needles from EtOH.Aq. M.p. 167–8°.

Schöpf, Braun, *Ann.*, 1934, 514, 69.

**Sambunigrin** (d-Glucoside of d-mandelic acid nitrile)

 $C_{14}H_{17}O_6N$ 

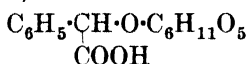
MW, 295

Occurs in black Holunder. Colourless cryst. from  $C_6H_6$ -amyl alcohol. M.p. 151–152.5°.  $[\alpha]_D^{18} - 75.1^\circ$ . Emulsin  $\rightarrow$  d-mandelonitrile + d-glucose.

*Tetra-acetyl*: needles from EtOH. M.p. 125–6°.  $[\alpha]_D^{22} - 52.5^\circ$ .

Fischer, Bergmann, *Ber.*, 1917, 50, 1063.

**Sambunigrinic Acid** ( $\beta$ -d-Glucoside of d-mandelic acid)

 $C_{14}H_{18}O_8$ 

MW, 314

Needles from amyl alcohol, needles + 1EtOH from EtOH, m.p. 175–7°. Sol.  $H_2O$ , EtOH, Py. Spar. sol.  $Et_2O$ ,  $Me_2O$ , AcOH,  $C_6H_6$ ,  $CHCl_3$ .  $[\alpha]_D^{18} + 51^\circ$  in  $H_2O$ .

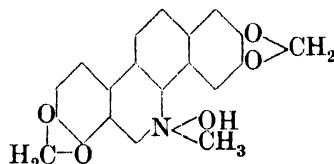
*Quinine salt*: prisms from  $H_2O$ . Sinters at 240°. M.p. 248° decomp.  $[\alpha]_D^{20} - 70.66^\circ$ .

*Me ester*:  $C_{15}H_{20}O_8$ . MW, 328. Needles from  $CHCl_3$ - $CCl_4$ . M.p. 88–9°. Sol.  $H_2O$ .  $[\alpha]_D^{19} + 41.2^\circ$ .

*Tetra-acetyl*: needles from EtOH.Aq. M.p. 166°.  $[\alpha]_D - 5.34^\circ$ .

Fischer, *Z. physiol. Chem.*, 1919, 107, 176; *Ber.*, 1919, 52, 200.

Karrer, Nägeli, Weidmann, *Helv. Chim. Acta*, 1919, 2, 259, 431.

Sanguinarine (*\psi*-Chelerythrine)

Suggested structure

 $C_{20}H_{15}O_5N$ 

MW, 349

Present in blood-root of *Sanguinaria canadensis*. Cryst. from  $Et_2O$ , m.p. 266° (242–3° slow heat.). Cryst. as alcoholate from alcoholic media, m.p. 195–7°. Sol. most org. solvents, sols. showing bluish-violet fluor.

Bruchhausen, Bersch, *Ber.*, 1930, 63, 2520.

Späth, Kuffner, *Ber.*, 1931, 64, 370, 1123, 2034.

## Sanguisorbigenin

 $C_{30}H_{46}O_3$ 

MW, 454

M.p. 275–6°. Heat  $\rightarrow$  sanguisorbigenol, a triterpene alcohol.

*Me ester*:  $C_{31}H_{48}O_3$ . MW, 468. M.p. 207–9°. *Acetyl*: m.p. 243–5°.

*Acetyl*: m.p. 324°.

Matsukawa, *Chem. Abstracts*, 1935, 29, 3346.

## Santal

 $C_{13}H_{10}O_4$ 

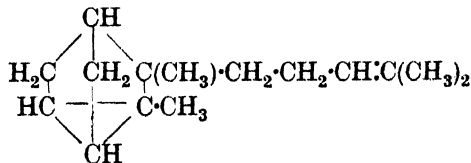
MW, 230

Plates or needles from EtOH. M.p. 222–3°. Sol. NaOH.  $FeCl_3 \rightarrow$  reddish-violet col.

*Di-Me ether*:  $C_{15}H_{14}O_4$ . MW, 258. M.p. 141°.

*Diacetyl*: colourless cryst. from MeOH. M.p. 152°.

Raudnitz, Perlmann, *Ber.*, 1935, 68, 1874.  
O'Neill, Perkin, *J. Chem. Soc.*, 1918, 113, 137.

 $\alpha$ -Santalene $C_{15}H_{24}$ 

MW, 204

Constituent of sandalwood oil. B.p. 252°/753 mm., 118–19°/9 mm.  $D_{20}^{20} 0.8984$ .  $n_D^{20} 1.491$ .  $[\alpha]_D - 15^\circ$ . S  $\rightarrow$  eudalene.

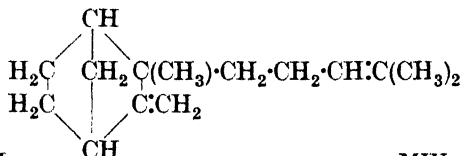
*Dihydrochloride*: b.p. 141–2°/0.5 mm.  $D_{20}^{20} 1.076$ .  $n_D^{20} 1.4976$ .  $[\alpha]_D + 6^\circ$ .



*Nitrosochloride*: m.p. 112–17°.

Semmler, *Ber.*, 1910, **43**, 1898; 1907, **40**, 3322.

Schimmel, *Chem. Zentr.*, 1910, II, 1757.

 **$\beta$ -Santalene**

$\text{C}_{15}\text{H}_{24}$  MW, 204

Constituent of sandalwood oil. Oil with cedar-like odour. B.p. 125–7°/9 mm.  $D_{20}^{25}$  0.894.  $n_D^{20}$  1.4946.  $[\alpha]_D - 35^\circ$ .

*Dihydrochloride*: same as from  $\alpha$ -santalene (above).

*Nitrosochloride*: two forms. (i) Plates. M.p. 152°. (ii) Needles. M.p. 106°.

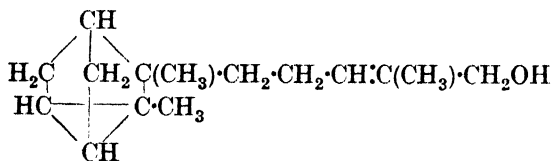
See previous references.

 **$\gamma$ -Santalene**

$\text{C}_{15}\text{H}_{24}$  MW, 204

Liq. B.p. 118°/10 mm.  $D_{20}^{20}$  0.9355.  $n_D$  1.5042.

Semmler, Bode, *Ber.*, 1907, **40**, 1130.

 **$\alpha$ -Santalol**

$\text{C}_{15}\text{H}_{24}\text{O}$  MW, 220

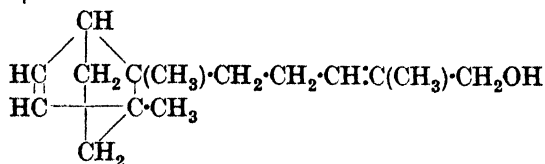
Constituent of sandalwood oil. B.p. 166–7°/14 mm., 106°/0.06 mm.  $D_{25}^{25}$  0.9770.  $n_D^{25}$  1.5017.  $\alpha_{5461} + 10.3^\circ$ .

*Allophanate*: needles from MeOH. M.p. 162–3°.

*Strychnine salt of hydrogen phthalate*: prisms from AcOEt or Me<sub>2</sub>CO. M.p. 144–5°.  $[\alpha]_{5461} - 5.62^\circ$  in C<sub>6</sub>H<sub>6</sub>.

Bradfield, Penfold, Simonsen, *J. Chem. Soc.*, 1935, 309.

Ruzicka, Thomann, *Helv. Chim. Acta*, 1935, **18**, 357.

 **$\beta$ -Santalol**

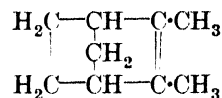
$\text{C}_{15}\text{H}_{24}\text{O}$  MW, 220

Constituent of sandalwood oil. B.p. 177–8°/17 mm., 112°/0.06 mm.  $D_{25}^{25}$  0.9717.  $n_D^{25}$  1.5100.  $\alpha_{5461} - 87.1^\circ$ .

*Allophanate*: needles from MeOH. M.p. 159–60°.

*Strychnine salt of hydrogen phthalate*: prisms from AcOEt. M.p. 134–5°.  $[\alpha]_{5461} - 37.5^\circ$  in C<sub>6</sub>H<sub>6</sub>.

See previous references.

**Santene**

$\text{C}_9\text{H}_{14}$  MW, 122

Constituent of sandalwood oil and pine-needle oil. Liq. with odour resembling camphene. B.p. 140–1°, 35°/15 mm.  $D_4^{17}$  0.8698.  $n_D^{17}$  1.4688.

*Nitrosite*: three forms. (i) Blue. M.p. 122–4°. (ii) Colourless. M.p. 104°. (iii) Green. M.p. 127–8° decomp.

*Nitrosate*: m.p. 216° decomp.

*Nitrosochloride*: blue. M.p. 109–10°. A white cryst. polymer is also described.

Diels, Alder, *Ann.*, 1931, **486**, 209.

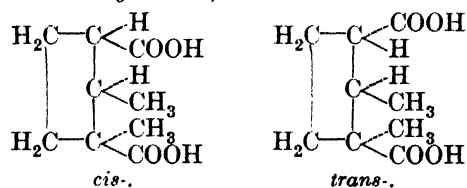
Ruzicka, Liebl, *Helv. Chim. Acta*, 1923, **6**, 271.

Komppa, Hintikka, *Bull. soc. chim.*, 1917, **21**, 14.

**Santene Hydrate.**

See  $\beta$ -Santenol.

**Santenic Acid** (1 : 2-Dimethylcyclopentane-1 : 3-dicarboxylic acid)



Probable configurations

$\text{C}_9\text{H}_{14}\text{O}_4$  MW, 186

*Cis*:

Acid from natural sources, by oxidation of santenol or santenone. Plates from H<sub>2</sub>O. M.p. 170–1°. AcOH-HCl  $\rightarrow$  *trans*-form.

*Di-Me ester*:  $\text{C}_{11}\text{H}_{18}\text{O}_4$ . MW, 214. B.p. 120–3°/9 mm.  $D_{20}^{20}$  1.078.  $n_D^{20}$  1.4645.

*Di-Et ester*:  $\text{C}_{13}\text{H}_{22}\text{O}_4$ . MW, 242. B.p. 143–4°/14 mm.  $D_{20}^{20}$  1.0268.  $n_D^{20}$  1.44854.

*Anhydride*:  $\text{C}_9\text{H}_{12}\text{O}_3$ . MW, 168. Cryst. M.p. 115–16°.

*Anil*:  $\text{C}_{15}\text{H}_{17}\text{O}_2\text{N}$ . MW, 243. Cryst. from AcOH.Aq. M.p. 117–18°.

**Monoanilide**: cryst. from AcOH.Aq. M.p. 205–6°.

**Trans**:

Cryst. from H<sub>2</sub>O. M.p. 166–7°.

**Dianilide**: needles from EtOH–C<sub>6</sub>H<sub>6</sub> or EtOH.Aq. M.p. 221°.

Komppa, *Chem. Zentr.*, 1936, I, 3838; *Ber.*, 1932, **65**, 1708.

Enkvist, *J. prakt. Chem.*, 1933, **137**, 261.

Komppa, Rohrmann, *Ber.*, 1934, **67**, 828.

Komppa, Hintikka, *Bull. soc. chim.*, 1917, **21**, 17.

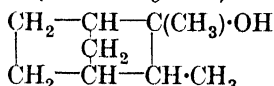
### Santenol.

See under Norborneol.

### α-Santenol.

See under Norborneol.

### β-Santenol (Santene hydrate)

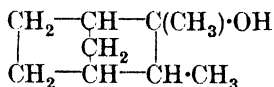


C<sub>9</sub>H<sub>16</sub>O MW, 140

Needles from EtOH. M.p. 101–2°. B.p. 192°. Stereoisomeric with γ-santenol.

Diels, Alder, *Ann.*, 1931, **486**, 205.

### γ-Santenol



C<sub>9</sub>H<sub>16</sub>O MW, 140

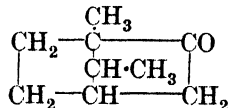
M.p. 63–5°. B.p. 82–4°/15 mm. Stereoisomeric with β-santenol.

Diels, Alder, *Ann.*, 1931, **486**, 209.

### Santenone.

See under Norcamphor.

### α-Santenone



C<sub>9</sub>H<sub>14</sub>O MW, 138

M.p. 55°. B.p. 191°.

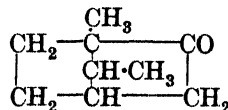
**Oxime**: m.p. 74°.

**Semicarbazone**: m.p. 236°.

Aschan, *Chem. Abstracts*, 1934, **28**, 2344.

Asahina, Ishidate, *Ber.*, 1935, **68**, 950.

### β-Santenone



C<sub>9</sub>H<sub>14</sub>O MW, 138

M.p. 46°. B.p. 190°.

**Oxime**: m.p. 51°.

**Semicarbazone**: m.p. 222°.

See previous references.

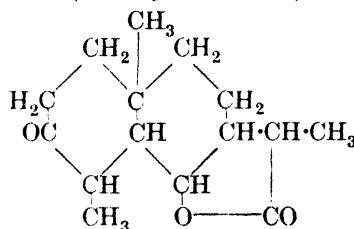
### Santenone Alcohol.

See under Norborneol.

### Santenylamine.

See under Norbornylamine.

### Santonan (Tetrahydrosantonin)



C<sub>15</sub>H<sub>22</sub>O<sub>3</sub>

MW, 250

Two forms.

α-

Cryst. from EtOH. M.p. 158°. [α]<sub>D</sub><sup>18</sup> + 17.8° in MeOH.

**Oxime**: cryst. from EtOH. M.p. 219–20°.

**Semicarbazone**: powder. M.p. 258° decomp.

**Phenylhydrazone**: cryst. from EtOH. M.p. 205° decomp.

β-

Plates from Et<sub>2</sub>O. M.p. 105°. [α]<sub>D</sub><sup>18</sup> + 9.3° in MeOH. More sol. than α-form.

**Oxime**: m.p. 182°.

**Semicarbazone**: m.p. 250° decomp.

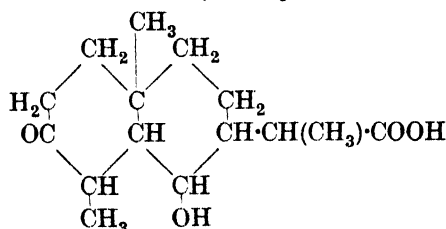
Clemo, Haworth, *J. Chem. Soc.*, 1930, 2580.

Wedekind, Goost, Jäckh, *Ber.*, 1930, **63**, 50.

Wienhaus, Oettingen, *Ann.*, 1913, **397**, 240.

Asahina, *Ber.*, 1913, **46**, 1776.

### Santonanic Acid (Tetrahydrosantoninic acid)



C<sub>15</sub>H<sub>24</sub>O<sub>4</sub>

MW, 268

α-

Cryst. + 1H<sub>2</sub>O. Sinters at 85°. M.p. 115° decomp. [α]<sub>D</sub><sup>18</sup> + 20.0°. Heat above m.p. → α-lactone.

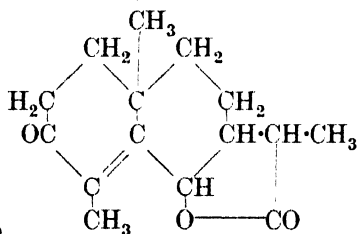
$\beta$ -.

Cryst. from  $H_2O$ . M.p. about  $192^\circ$ .  $[\alpha]_D^{18} + 2.2^\circ$ . Heat above m.p.  $\rightarrow$   $\beta$ -lactone.

Oxime: m.p.  $218-20^\circ$ .

Wienhaus, Oettingen, *Ann.*, 1913, **397**, 238.

### Santonene (Dihydrosantonin)



$C_{15}H_{20}O_3$  MW, 248

Two forms.

(i) Prisms or needles from EtOH.Aq. M.p.  $105^\circ$ .  $[\alpha]_D^{18} + 75.3^\circ$  in EtOH.

Oxime: prisms from EtOH. M.p.  $235^\circ$  decomp.

Semicarbazone: prisms from EtOH. M.p.  $245^\circ$  decomp.

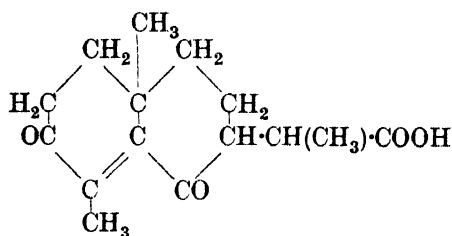
(ii) Needles from EtOH. M.p.  $181-2^\circ$ .  $[\alpha]_D^{25} - 280.9^\circ$ . Insol.  $Na_2CO_3$ .

Oxime: needles. M.p.  $196^\circ$ .  $[\alpha]_D^{25} - 239^\circ$  in EtOH.

Wedekind, Goost, Jäckh, *Ber.*, 1930, **63**, 53.

Medvedev, Alekseeva, *Chem. Abstracts*, 1928, **22**, 1979.

### Santonin



$C_{15}H_{18}O_3$  MW, 246

Cryst. from  $H_2O$  or EtOH. M.p.  $170-2^\circ$ . B.p.  $285/15$  mm.  $[\alpha]_D^{20} - 74.0^\circ$  in  $CHCl_3$ . Sol. 190 parts  $H_2O$  at  $17^\circ$ . Dist.  $\rightarrow$  meta-santonin acid.  $H_2SO_4 \rightarrow$  santonin.

Me ester:  $C_{16}H_{22}O_4$ . MW, 278. Prisms from MeOH, needles from EtOH.Aq. M.p.  $86^\circ$ .  $[\alpha]_D^{20} - 52.3^\circ$  in  $CHCl_3$ . Oxime: prisms. M.p.  $158-9^\circ$ .

Et ester:  $C_{17}H_{24}O_4$ . MW, 292. Prisms from Et<sub>2</sub>O. M.p.  $94-5^\circ$ .  $[\alpha]_D^{20} - 45.4^\circ$  in  $CHCl_3$ . Oxime: plates from EtOH. M.p.  $126^\circ$ . Monophenylhydrazone: yellow plates. M.p.  $115^\circ$ .

Propyl ester:  $C_{18}H_{26}O_4$ . MW, 306. Syrup. B.p.  $220/3$  mm.

Isobutyl ester:  $C_{19}H_{28}O_4$ . MW, 320. Needles from Et<sub>2</sub>O. M.p.  $67^\circ$ .

Chloride:  $C_{15}H_{19}O_3Cl$ . MW, 282.5. Prisms from EtOH. M.p.  $170-1^\circ$ .

Bromide:  $C_{15}H_{19}O_3Br$ . MW, 327. M.p.  $145.5^\circ$ .

Iodide:  $C_{15}H_{19}O_3I$ . MW, 374. Needles from Et<sub>2</sub>O. M.p.  $136^\circ$ .

Oxime: cryst. from EtOH. M.p.  $186^\circ$ .

Dioxime: m.p.  $120-5^\circ$  decomp.

Monosemicarbazone: m.p.  $183-5^\circ$  decomp.

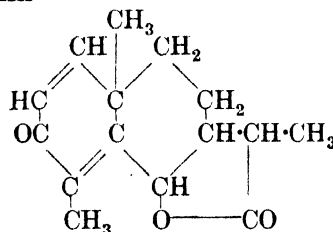
Monophenylhydrazone: yellow needles from EtOH. M.p.  $174^\circ$ .

Abkin, Medvedev, *Chem. Abstracts*, 1935, **29**, 3682.

Harries, Stähler, *Ber.*, 1904, **37**, 259.

Francesconi, *Gazz. chim. ital.*, 1899, **29**, 224.

### Santonin



$C_{15}H_{18}O_3$  MW, 246

Widely occurring in plants, especially in *Artemisia*. Colourless cryst. from  $H_2O$ , EtOH or Et<sub>2</sub>O. M.p.  $170^\circ$ .  $[\alpha]_D^{18} - 173.0^\circ$  in EtOH. Turns yellow in sunlight. Isomerises in sol. in daylight.  $H_2SO_4 \rightarrow$  blue col. EtOH-KOH  $\rightarrow$  red col. Zn + AcOH  $\rightarrow$  santonone. Zn dust dist.  $\rightarrow$  1:4-dimethylnaphthalene and 1:4-dimethyl-2-naphthol. Alkalis  $\rightarrow$  salts of santonin acid. Sol. has bitter taste. Used in treatment of nervous complaints.

Oxime: needles +  $1H_2O$  from EtOH.Aq. M.p.  $218^\circ$ .

Semicarbazone: cryst. from AcOEt. M.p.  $232^\circ$  decomp.

Phenylhydrazone: yellow needles. M.p.  $230-1^\circ$  decomp.

Clemons, Haworth, *J. Chem. Soc.*, 1930, 2579.

Ruzicka, Eichenberger, *Helv. Chim. Acta*, 1930, **13**, 1117.

Wedekind, Tettweiler, *Ber.*, 1931, **64**, 1796.

Massagetov, *Chem. Abstracts*, 1932, **26**, 4413.

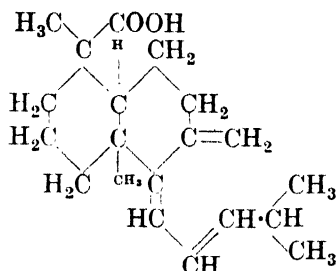
**$\beta$ -Santonin** $C_{15}H_{18}O_3$ 

MW, 246

Stereoisomeric with santonin. Constituent of certain varieties of *Artemisia*. Colourless prisms from EtOH. M.p. 216–18°.  $[\alpha]_D^{19} - 137.2^\circ$  in  $CHCl_3$ .

Oxime : prisms from EtOH. M.p. 224°.

Clemons, *J. Chem. Soc.*, 1934, 1343.

**1-Säpietic Acid (1-Pimaric acid)**

Suggested structure

 $C_{20}H_{30}O_2$ 

MW, 302

Occurs in *Pinus palustris*, etc. Prisms from EtOH. M.p. 152° (148–51°).  $[\alpha]_D^{20} - 282^\circ$  in EtOH. Isomerised by hot AcOH to abietic acid. Absorption maximum at 272.5 m $\mu$ .

Me ester :  $C_{21}H_{32}O_2$ . MW, 316. M.p. 57°. B.p. 166–9°/0.5 mm.  $D_4^{22} 1.0312$ .  $n_D^{23} 1.5232$ .  $[\alpha]_D - 190.4^\circ$  in EtOH.

Et ester :  $C_{22}H_{34}O_2$ . MW, 330. B.p. 175–7°/0.5 mm.  $D_4^{23} 1.0124$ .  $n_D^{23} 1.5153$ .  $[\alpha]_D - 170.9^\circ$  in EtOH.

Hasselstrom, Bogert, *J. Am. Chem. Soc.*, 1935, 57, 2118.

Palkin, Morris, *J. Am. Chem. Soc.*, 1933, 55, 3677.

Ruzicka, Balos, Vilim, *Helv. Chim. Acta*, 1924, 7, 458.

**Saponarin (Vitexin glucoside)** $C_{21}H_{24}O_{12}$ 

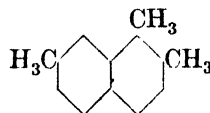
MW, 468

Occurs in *Saponaria officinalis*, Linn., and other plants. Needles + 2H<sub>2</sub>O from Py.Aq. M.p. 231–2° decomp. (slow heat), 236° (rapid heat).  $[\alpha]_D - 7.9^\circ$  in H<sub>2</sub>O. Sol. alkalis with yellow col. Conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  blue fluor. I  $\rightarrow$  blue col. which disappears on dilution or addition of EtOH or on warming; reappears on cooling.

Nona-acetyl deriv. : m.p. 183–5°.

Molisch, *Chem. Abstracts*, 1912, 6, 766.

Barger, Field, *J. Chem. Soc.*, 1912, 101, 1396.

**Sapotalin (1 : 2 : 7-Trimethylnaphthalene)** $C_{13}H_{14}$ 

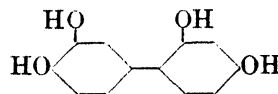
MW, 170

B.p. 148°/16 mm.  $D_4^{15} 1.008$ .  $n_D^{15} 1.6093$ .

Picrate : m.p. 129.5–130°.

Styphnate : m.p. 156°.

Spring, Vickerstaff, *J. Chem. Soc.*, 1937, 252.

**Sappanin (2 : 4 : 3' : 4'-Tetrahydroxydiphenyl)** $C_{12}H_{10}O_4$ 

MW, 218

Occurs in *Caesalpinia Sappan*, Linn. Leaflets + 2H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 210–11°. B.p. 230–40°/0.01 mm. Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O. Insol. CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. FeCl<sub>3</sub>  $\rightarrow$  red. col. Reduces Fehling's and Tollen's on warming.

Tetra-Me ether :  $C_{16}H_{18}O_4$ . MW, 274. Cryst. from MeOH.Aq. M.p. 74–5°.

Späth, Gibian, *Monatsh.*, 1930, 55, 342.

**Sarcine.**

See Hypoxanthine.

**Sarcosine.**

See under Lactic Acid.

**Sarcosine** (Methylaminoacetic acid, N-methylglycine, N-methylglycocol)

 $C_3H_7O_2N$ 

MW, 89

Cryst. from EtOH. M.p. 212–13° decomp. Heat of comb. C<sub>v</sub> 401.2 Cal.

B.HCl : m.p. 168–70°.

B.HBr : m.p. 186–7°.

B.HI : m.p. 152°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub> : m.p. 193–4° decomp.

Et ester :  $C_5H_{11}O_2N$ . MW, 117. B.p. 43°/10 mm.  $D_4^{15} 0.971$ . Picrate : m.p. 149.5°.

Nitrile :  $C_3H_6N_2$ . MW, 70. Viscous liq.

B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub> : does not melt below 210°.

N-Benzoyl : N-methylhippuric acid. M.p. 103.5–104° decomp.

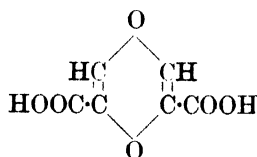
*N*-Phenyl: see *N*-Phenylsarcosine.

Baumann, *J. Biol. Chem.*, 1915, **21**, 563.

Cocker, Lapworth, *J. Chem. Soc.*, 1931, 1894.

Johnson, Ambler, *J. Am. Chem. Soc.*, 1914, **36**, 372.

### Sarsapic Acid (*Sarsapinic acid*)



Probable structure

$C_6H_4O_6$

MW, 172

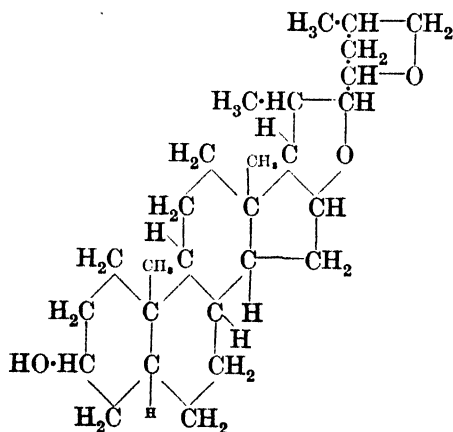
Occurs in sarsaparilla root. Needles from hot  $H_2O$ . M.p.  $305^\circ$ . Sol. EtOH. Spar. sol.  $Et_2O$ . *Di-Me ester*:  $C_8H_8O_6$ . MW, 200. Leaflets from EtOH. M.p.  $121^\circ$ .

Power, Salway, *J. Chem. Soc.*, 1914, **105**, 205.

### Sarsapinic Acid.

See Sarsapic Acid.

### Sarsasapogenin (*Parigenin*)



Suggested structure

$C_{27}H_{44}O_3$

MW, 416

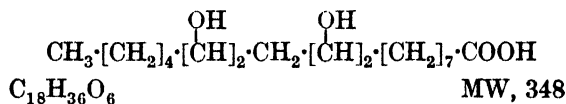
Occurs in Vera-Cruz sarsaparilla root. Cryst. from  $Me_2CO$ . M.p.  $197-8^\circ$ .  $[\alpha]_D^{25} - 75^\circ$  in  $CHCl_3$ .

*Acetyl*: m.p.  $144-5^\circ$ .  $[\alpha]_D^{25} - 70.2^\circ$  in  $CHCl_3$ .

*Benzoyl*: m.p.  $170-1^\circ$ .

Farmer, Kon, *J. Chem. Soc.*, 1937, 414.

### Sativic Acid (*Sativinic acid*, 8 : 9 : 11 : 12-tetrahydroxystearic acid)



$C_{18}H_{36}O_6$

MW, 348

Occurs in bark of various plants and in human fat. ( $\alpha$ ) M.p.  $153^\circ$ . ( $\beta$ ) M.p.  $170^\circ$ . ( $\gamma$ ) M.p.  $144-5^\circ$ . ( $\delta$ ) M.p.  $135^\circ$ .

Zellner, *Monatsh.*, 1925, **46**, 619.

Nicolet, Cox, *J. Am. Chem. Soc.*, 1922, **44**, 144.

Dreyfuss, F.P., 636,488, (*Chem. Abstracts*, 1929, **23**, 1140).

Wagner, *Biochem. Z.*, 1926, **174**, 412.

Reinger, *Chem. Abstracts*, 1922, **16**, 3467.

### Sativinic Acid.

See Sativic Acid.

### Saxatilic Acid.

See Salazinic Acid.

### Scammonin.

See Jalapin.

### Scatole.

See Skatole.

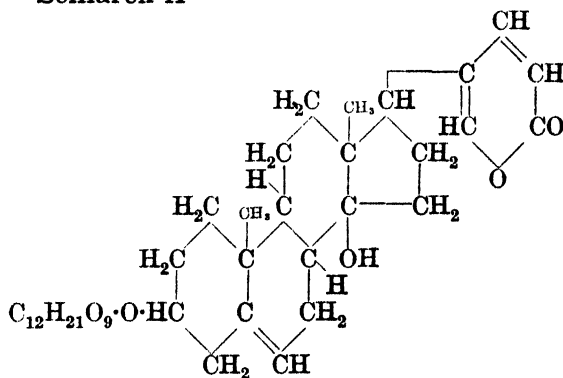
### Schäffer Acid.

See 2-Naphthol-6-sulphonic Acid.

### Schöllkopf Acid.

See 1-Naphthylamine-8-sulphonic Acid.

### Scillaren-A



Suggested structure

$C_{36}H_{52}O_{13}$

MW, 692

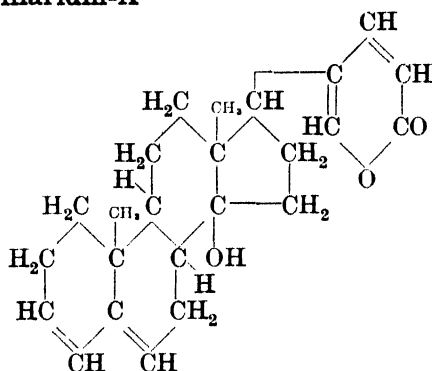
Occurs in squills. Plates +  $1H_2O$  +  $1MeOH$  from  $MeOH.Aq.$ , m.p.  $230-40^\circ$ ; needles from EtOH, m.p.  $270^\circ$ .  $[\alpha]_D^{20} - 73.8^\circ$  in EtOH.

Tschesche, Haupt, *Ber.*, 1937, **70**, 44.

Stoll, Hofmann, *Helv. Chim. Acta*, 1935, **18**, 82, 401.

Stoll *et al.*, *Helv. Chim. Acta*, 1933, **16**, 703; 1935, **18**, 649.

## Scillaridin-A



Suggested structure

$C_{24}H_{30}O_3$  MW, 366

Prisms from EtOH. M.p. 245–50°. Spar. sol. EtOH,  $CHCl_3$ . Insol.  $H_2O$ ,  $Et_2O$ .  $[\alpha]_D^{20}$  – 62.6° in  $MeOH-CHCl_3$ .

See first reference above and also Stoll *et al.*, *Helv. Chim. Acta*, 1933, 16, 727; 1935, 18, 647, 1247.

## Scoparin

$C_{22}H_{22}O_{11}$  MW, 462

Occurs in *Cytisus scoparius*, Link. M.p. 202–19° decomp. Sol. hot  $H_2O$ , EtOH. Insol.  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ .

*Di-Me ether*:  $C_{24}H_{26}O_{11}$ . MW, 490. Yellow cryst. M.p. 260–5° decomp.

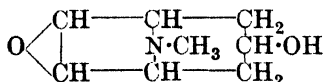
*Tri-Me ether*:  $C_{25}H_{28}O_{11}$ . MW, 504. Yellow cryst. M.p. 220–38°.

*Octa-Me ether*:  $C_{29}H_{36}O_{11}$ . MW, 560. M.p. 120–30° → solid, remelting at 229–33°.

Hemmelmayer, Strehly, *Monatsh.*, 1926, 47, 379.

Herzig, Tiring, *Monatsh.*, 1918, 39, 253.

## Scopine



$C_8H_{13}O_2N$  MW, 155

Needles from pet. ether. M.p. 76°.

$B,HAuCl_4$ : m.p. 216° decomp.

$B_2, H_2PtCl_6$ : m.p. 219° decomp.

*Picrate*: leaflets. M.p. 231°.

*Tropic ester*: see Hyoscyne.

$\psi$ -Scopine.

M.p. 125–6°.

*Benzoyl*: m.p. 142°.  $B,HCl$ : m.p. 216°.

*Chloroaurate*: m.p. 220°. *Picrate*: m.p. 204°.

*Phenylurethane*: m.p. 229°.  $B,HAuCl_4$ : m.p. 210°.

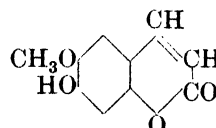
Willstätter, Berner, *Ber.*, 1923, 56, 1079, 1081.

Polonovski, Polonovski, *Bull. soc. chim.*, 1928, 43, 590, 594.

## Scopolamine.

See Hyoscyne.

**Scopoletin** (*Æsculetin* 6-methyl ether, *chrys-atropic acid*, *gelseminic acid*, 7-hydroxy-6-methoxy-coumarin,  $\beta$ -methylæsculetin)



$C_{10}H_8O_4$

MW, 192

Occurs in root of *Gelsemium sempervirens*, Ait., *Atropa belladonna*, Linn., *Convolvulus scammonia*, Linn., *Ipomoea orizabensis*, Ledenois, *Prunus serotina*, Ehrh. and *Fabiana imbricata*, Ruiz & Pav. Needles or prisms. M.p. 204°. Sol. hot EtOH, hot AcOH. Mod. sol.  $CHCl_3$ . Spar. sol.  $H_2O$ . Insol.  $C_6H_6$ ,  $CS_2$ . Blue fluor. in EtOH. Reduces Fehling's and Tollen's.  $FeCl_3$  → green col.

*Acetyl*: m.p. 177°.

*Me ether*: 6:7-dimethoxycoumarin.  $C_{11}H_{10}O_4$ . MW, 206. M.p. 145°.

*Glucoside*: see Fabiatriin.

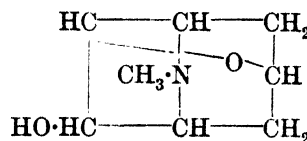
Seka, Kallir, *Ber.*, 1931, 64, 909 (*Bibl.*).

Head, Robertson, *J. Chem. Soc.*, 1931, 1241 (*Bibl.*).

## Scopoligenin.

See Norscopoline.

**Scopoline** (*Oscine*)



$C_8H_{13}O_2N$

MW, 155

Prisms from  $Et_2O$ . M.p. 108–9°. B.p. 248°.

$B,HCl$ : m.p. 270°.

$B,HAuCl_4$ : m.p. 220° decomp.

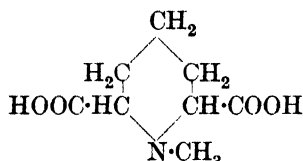
$B_2, H_2PtCl_6$ : m.p. 203° decomp.

*Picrate*: m.p. 236°.

Willstätter, Berner, *Ber.*, 1923, 56, 1079.

Polonovski, Polonovski, *Bull. soc. chim.*, 1928, 43, 590.

**Scopolinic Acid** (*N*-Methylhexahydrodipicolinic acid, *N*-methylpiperidine-2:6-dicarboxylic acid)



$C_8H_{13}O_4N$

MW, 187

Plates +  $1H_2O$ . M.p.  $225^\circ$  decomp. ( $230^\circ$  anhyd.). Sol. hot  $H_2O$ . Spar. sol.  $Et_2O$ .  $B.HCl$ : m.p.  $225-6^\circ$  decomp.

*Di-Me ester*:  $C_{10}H_{17}O_4N$ . MW, 215. B.p.  $140-1^\circ/13$  mm.

Schmidt, *Arch. Pharm.*, 1915, **253**, 606.

Hess, Wissing, *Ber.*, 1915, **48**, 1910.

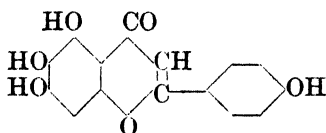
Willstätter, Lessing, *Ber.*, 1902, **35**, 2072.

Hess, Suchier, *Ber.*, 1915, **48**, 2057.

### Scopularic Acid.

See *Stictic Acid*.

**Scutellarein** (5:6:7:4'-Tetrahydroxyflavone)



$C_{15}H_{10}O_6$

MW, 286

Yellow leaflets from MeOH. Does not melt below  $300^\circ$ . Sol. boiling MeOH, EtOH, AcOH. Spar. sol. other solvents. Insol.  $H_2O$ . KOH  $\rightarrow$  reddish-yellow sol. Cold  $NH_3$ .  $AgNO_3$  gives reddish-brown col. and is reduced on heating. Alc.  $FeCl_3 \rightarrow$  reddish-brown col.

6:4'-*Di-Me ether*:  $C_{17}H_{14}O_6$ . MW, 314. Yellow cryst. M.p.  $219^\circ$ . *Diacetyl*: m.p.  $149-50^\circ$ .

6:7:4'-*Tri-Me ether*:  $C_{18}H_{16}O_6$ . MW, 328. Yellow microleaflets from AcOEt. M.p.  $189-90^\circ$ . Conc.  $H_2SO_4 \rightarrow$  greenish-yellow sol. *Acetyl*: microleaflets from AcOEt. M.p.  $169^\circ$ .

*Tetra-Me ether*:  $C_{18}H_{18}O_6$ . MW, 342. Exists in two forms. (i) Prisms from EtOH. M.p.  $161^\circ$ . (ii) Cryst. from EtOH. M.p.  $142^\circ$ .

*Tetra-acetyl*: needles from AcOEt or EtOH. M.p.  $235-7^\circ$ .

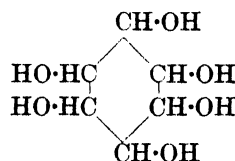
Bargellini, *Gazz. chim. ital.*, 1915, **45**, i, 77.

Goldschmiedt, Zerner, *Monatsh.*, 1910, **31**, 464.

Robinson, Schwarzenbach, *J. Chem. Soc.*, 1930, 829.

Wessely, Moser, *Monatsh.*, 1930, **56**, 97.

**Scyllitol** (*Cocosite*, *cocositol*, *quercin*, *quercinite*, *hexahydroxycyclohexane*, stereoisomer of *inositol*)



$C_6H_{12}O_6$

MW, 180

Occurs in dogfish (*Scyllium canicula*) and other fish; in *Acanthus vulgaris*, *Cornus florida*, Linn. (flowering dogwood) and various *Cocos* species. Prisms +  $3H_2O$  from  $H_2O$ . M.p.  $353^\circ$  decomp. Spar. sol.  $H_2O$ . Insol. EtOH,  $Et_2O$ , MeOH,  $C_6H_6$ ,  $CHCl_3$ .

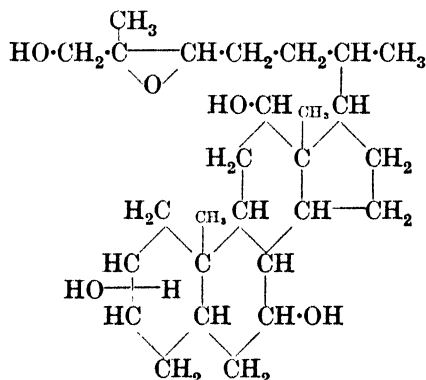
*Hexa-acetyl*: m.p.  $291^\circ$ .

Needham, *Biochem. J.*, 1929, **23**, 319.

Hann, Sando, *J. Biol. Chem.*, 1926, **68**, 399.

Goodson, *J. Chem. Soc.*, 1920, **117**, 140.

### Scymnol



$C_{27}H_{46}O_5$

MW, 450

Occurs as sulphuric ester in bile of shark (*Scymnus borealis*). Cryst. +  $2H_2O$  from  $Me_2CO$ . Aq., m.p.  $115^\circ$ : anhyd. cubes from AcOEt, m.p.  $187^\circ$ .  $[\alpha]_D + 38.2^\circ$  in EtOH.

*Hydrochloride*: two forms. (i) Needles from MeOH. M.p.  $196^\circ$ . (ii) M.p.  $126^\circ$ . Both forms give scymnol with alkalis.

*Tetra-acetyl*: plates from MeOH. M.p.  $148^\circ$ . B.p.  $280^\circ/0.005$  mm.

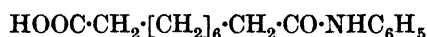
Tschesche, *Z. physiol. Chem.*, 1931, **203**, 263.

Windaus, Bergmann, König, *Z. physiol. Chem.*, 1930, **189**, 148.

Oikawa, *Chem. Abstracts*, 1926, **20**, 401.

### Sebacamic Acid.

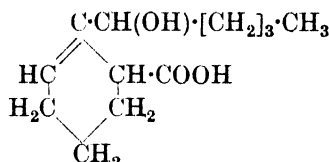
See under *Sebacic Acid*.

**Sebacanilic Acid** (*Sebacic acid monoanilide*) $\text{C}_{16}\text{H}_{23}\text{O}_3\text{N}$ 

MW, 277

Leaflets from  $\text{H}_2\text{O}$ . M.p. 121–2°.*Me ester*:  $\text{C}_{17}\text{H}_{25}\text{O}_3\text{N}$ . MW, 291. Needles from pet. ether. M.p. 67–8°.Morgan, Walton, *J. Chem. Soc.*, 1936, 905.**Sebacic Acid** (*Octane-1 : 8-dicarboxylic acid*) $\text{C}_{10}\text{H}_{18}\text{O}_4$ 

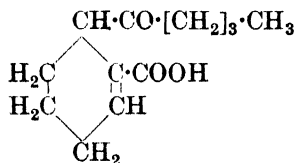
MW, 202

Leaflets. M.p. 134.5° (132–3°). B.p. 294.5°/100 mm., 273°/50 mm., 243.5°/15 mm., 232°/10 mm. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Heat of comb.  $C_p$  1297.3 Cal.,  $C_v$  1295.9 Cal.  $k$  (first) =  $2.6 \times 10^{-5}$  at 25°; (second) =  $2.6 \times 10^{-6}$  at 100°.*Me ester*:  $\text{C}_{11}\text{H}_{20}\text{O}_4$ . MW, 216. M.p. 40–1°. B.p. 208°/20 mm. *Chloride*:  $\text{C}_{11}\text{H}_{19}\text{O}_3\text{Cl}$ . MW, 234.5. B.p. 177°/23 mm.*Di-Me ester*:  $\text{C}_{12}\text{H}_{22}\text{O}_4$ . MW, 230. M.p. 26.4° (38°). B.p. 293°/754 mm., 175°/20 mm.  $D_4^{25}$  0.98818.  $n_D^{25}$  1.43549.*Et ester*:  $\text{C}_{12}\text{H}_{22}\text{O}_4$ . MW, 230. M.p. 35°. B.p. 202–3°/15 mm.  $k = 1.43 \times 10^{-5}$  at 25°.*Di-Et ester*:  $\text{C}_{14}\text{H}_{26}\text{O}_4$ . MW, 258. M.p. 1.25°. B.p. 306°/773 mm., 158–9°/7.5 mm.  $D_4^{20}$  0.96461.  $n_D^{20}$  1.43589.*Dibutyl ester*:  $\text{C}_{18}\text{H}_{34}\text{O}_4$ . MW, 314. B.p. 344–5°.  $D^{15}$  0.9329.*Di-isoamyl ester*:  $\text{C}_{20}\text{H}_{38}\text{O}_4$ . MW, 342. M.p. 18°. B.p. 202–3°/2–3 mm.  $D_4^{25}$  0.9230.*Glycol ester*:  $\text{C}_{12}\text{H}_{20}\text{O}_4$ . MW, 228. M.p. 79°.*Trimethylene glycol ester*:  $\text{C}_{13}\text{H}_{22}\text{O}_4$ . MW, 242. M.p. 14–17°. B.p. 130–3°/2 mm.  $D_4^{20}$  1.0747.  $n_D^{20}$  1.4719.*Di-p-nitrobenzyl ester*:  $\text{C}_{24}\text{H}_{28}\text{O}_6\text{N}_2$ . MW, 472. M.p. 72–6°.*Dimenthyl ester*:  $\text{C}_{30}\text{H}_{54}\text{O}_4$ . MW, 478. B.p. 256–8°/20 mm.*Diphenacyl ester*:  $\text{C}_{26}\text{H}_{25}\text{O}_4$ . MW, 404. M.p. 80–4°.*Dichloride*:  $\text{C}_{10}\text{H}_{16}\text{O}_2\text{Cl}_2$ . MW, 239. B.p. 185–95°/30 mm., 182°/16 mm.  $n_D^{18.3}$  1.46836.*Monoamide*: sebacamic acid.  $\text{C}_{10}\text{H}_{19}\text{O}_3\text{N}$ . MW, 201. M.p. 170°.*Diamide*:  $\text{C}_{10}\text{H}_{20}\text{O}_2\text{N}_2$ . MW, 200. M.p. 210° (208°).*Dinitrile*: octamethylene dicyanide, sebaco-nitrile.  $\text{C}_{10}\text{H}_{16}\text{N}_2$ . MW, 164. B.p. 199–200°/15 mm.*Anhydride*:  $\text{C}_{10}\text{H}_{16}\text{O}_3$ . MW, 184. Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 78–9°.*Monoanilide*: see Sebacanilic Acid.*Dianilide*: m.p. 198°.*Dihydrazide*: m.p. 184–5°.Morgan, Walton, *J. Chem. Soc.*, 1936, 903.Izard, U.S.P., 1,991,391, (*Chem. Abstracts*, 1935, 29, 2176).Spanagel, F.P., 796,410, (*Chem. Abstracts*, 1936, 30, 6138).Stoll, Rouvé, *Helv. Chim. Acta*, 1936, 19, 253.Fischl, Steiner, U.S.P., 1,876,652, (*Chem. Abstracts*, 1933, 27, 102).Naegeli, Münzel, D.R.P., 554,700, (*Chem. Abstracts*, 1932, 26, 5970).Kao, Ma, *Chem. Abstracts*, 1932, 26, 4305.Landa, Kejvan, *Chem. Abstracts*, 1932, 26, 78.Montonna, *J. Am. Chem. Soc.*, 1927, 49, 2114.Boedtker, *Chem. Abstracts*, 1924, 18, 3043.**Sedanolic Acid** $\text{C}_{12}\text{H}_{20}\text{O}_3$ 

MW, 212

Needles from  $\text{C}_6\text{H}_6$ . M.p. 88–9°. Sol. Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ , hot pet. ether. Insol. H<sub>2</sub>O.*Lactone*: sedanolide.  $\text{C}_{12}\text{H}_{18}\text{O}_2$ . MW, 194. Occurs in celery oil. B.p. 185°/17 mm.  $D_4^{25}$  1.0383.  $n_D^{25.5}$  1.4923.  $[\alpha]_D^{25.5} = 23.66^\circ$ .Ciamician, Silber, *Ber.*, 1897, 30, 497.**Sedanolide.**

See under Sedanolic Acid.

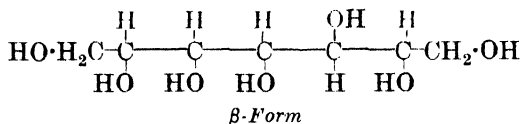
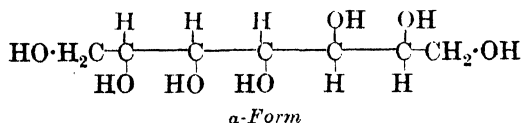
**Sedanonic Acid** $\text{C}_{12}\text{H}_{18}\text{O}_3$ 

MW, 210

Saponification product from celery oil. Needles from  $\text{C}_6\text{H}_6$ . M.p. 113°. Sol. EtOH, AcOH. Mod. sol. Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ . Spar. sol. pet. ether. Insol. H<sub>2</sub>O.*Oxime*: m.p. 128°.Ciamician, Silber, *Ber.*, 1897, 30, 500.



## Sedoheptitol

 $\text{C}_7\text{H}_{16}\text{O}_7$ 

MW, 212

*α-Form*: Volemitol, *d*- $\beta$ -Mannoheptitol.

Occurs in *Lactarius volemus*, etc. Needles from dil. EtOH. M.p. 154–5° (151–2°).  $[\alpha]_D^{20} + 2.25^\circ$  in  $\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol. Et<sub>2</sub>O.

*Triethylidene deriv.*: cryst. M.p. 191–4°.  $[\alpha]_D - 45.55^\circ$  in  $\text{CHCl}_3$ ,  $[\alpha]_D - 72.35^\circ$  in  $\text{CHCl}_3$ , – 117.6° in Py.

*Tribenzylidene deriv.*: needles. M.p. 214–15° (199–200°).  $[\alpha]_D^{20} - 1.7^\circ$  in  $\text{CHCl}_3$ , – 48.4° in Py.

*β-Form*:

Plates or needles from dil. EtOH. M.p. 127–8°.

*Tribenzylidene deriv.*: m.p. 272–5°.

Bougault, Allard, *Compt. rend.*, 1902, **135**, 796.

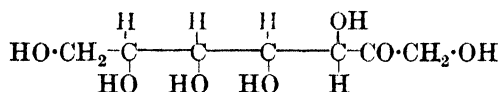
La Forge, Hudson, *J. Biol. Chem.*, 1917, **30**, 68; 1928, **79**, 1.

La Forge, *J. Biol. Chem.*, 1920, **42**, 375.

Ettel, *Chem. Zentr.*, 1929, II, 714.

Ettel, *Chem. Abstracts*, 1933, **27**, 1617.

## Sedoheptose

 $\text{C}_7\text{H}_{14}\text{O}_7$ 

MW, 210

Occurs in *Sedum spectabile*. Syrup. Weakly dextrorotatory. Reduces Fehling's.  $\text{NaHg} \rightarrow \alpha + \beta$ -sedoheptitol.

*Phenylosazone*: cryst. M.p. 197° decomp.

*p*-*Bromophenylosazone*: yellow needles. M.p. 227–8° decomp.

La Forge, *J. Biol. Chem.*, 1920, **42**, 367.

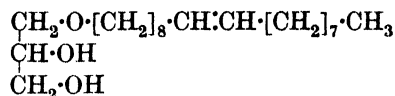
La Forge, Hudson, *J. Biol. Chem.*, 1917, **30**, 61.

See also last reference above.

## Selacholeic Acid.

See Nervonic Acid.

**Selachyl Alcohol** (*Glycerol 1-octadecenyl ether, α-oleyl glyceryl ether*)

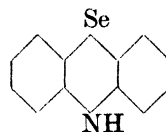
 $\text{C}_{21}\text{H}_{42}\text{O}_3$ 

MW, 342

Occurs in liver oil of *Centrophorus granulosus*. B.p. 242°/5 mm.  $D_4^{15} 0.9206$ .  $n_D^{15} 1.4690$ .

Davies, Heilbron, Jones, *J. Chem. Soc.*, 1933, 165.

**Selenazine** (*Phenoselenazine, selenodiphenylamine*)

 $\text{C}_{12}\text{H}_9\text{NSe}$ 

MW, 246

Yellow leaflets from  $\text{C}_6\text{H}_6$ . M.p. 195°. Sol. ord. org. solvents. Oxidises in air.

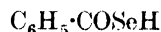
*N-Acetyl*: m.p. 176°.

*N-Me*:  $\text{C}_{13}\text{H}_{11}\text{NSe}$ . MW, 260. M.p. 138–9°.

Cornelius, *Chem.-Ztg.*, 1913, **37**, 198.

Weizmann, Stephen, *Proc. Chem. Soc.*, 1913, **29**, 196.

## Selenobenzoic Acid

 $\text{C}_7\text{H}_6\text{OSe}$ 

MW, 185

Red cryst. from MeOH. M.p. 133°.

*p*-*Tolyl ester*:  $\text{C}_{14}\text{H}_{12}\text{OSe}$ . MW, 275. Prisms from EtOH. M.p. 71–2°. Turns red in air.

*p*-*Methoxyphenyl ester*:  $\text{C}_{14}\text{H}_{12}\text{O}_2\text{Se}$ . MW, 291. Cryst. from EtOH. M.p. 97°.

*p*-*Ethoxyphenyl ester*:  $\text{C}_{15}\text{H}_{14}\text{O}_2\text{Se}$ . MW, 305. Needles from ligroin. M.p. 97°.

*Amide*:  $\text{C}_7\text{H}_7\text{NSe}$ . MW, 184. Golden needles from Et<sub>2</sub>O. M.p. 126° (115°).

Mingoia, *Gazz. chim. ital.*, 1926, **56**, 835.

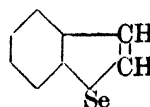
Becker, Meyer, *Ber.*, 1904, **37**, 2550.

## Selenodiphenylamine.

See Selenazine.

## Selenofuran.

See Selenophene.

Selenonaphthene (*Benzselenophene*) $\text{C}_8\text{H}_6\text{Se}$ 

MW, 181



- 1-Chloroacetyl : m.p. 111–12°.  
 1-Dichloroacetyl : m.p. 108°.  
 1-Trichloroacetyl : m.p. 154° decomp.  
 1-Benzoyl : m.p. 225°.

Audrieth, *J. Am. Chem. Soc.*, 1930, **52**, 1250.

Mistry, Guha, *J. Indian Chem. Soc.*, 1930, **1**, 793.

Ingersoll, Bircher, Brubaker, *Organic Syntheses*, Collective Vol. I, 472.

### 3-Semicarbazidobenzamide.

See Kryogenin.

#### Seminose.

See Mannose.

#### Semioxamazide.

See under Oxamic Acid.

#### Sempervine.

See Sempervirine.

#### Sempervirine (Sempervine)

$C_{19}H_{16}N_2$  MW, 272

Occurs in *Gelsemium sempervirens*, Ait. Cryst. +  $H_2O$ , m.p. 258–60°: cryst. from  $CHCl_3$ , m.p. 223°: cryst. from EtOH, m.p. 254°. Sol. EtOH,  $CHCl_3$ , Py. Insol.  $Et_2O$ ,  $C_6H_6$ .

Hasenfratz, *Compt. rend.*, 1933, **196**, 1530.

Chou, *Chem. Abstracts*, 1931, **25**, 5736.

Stevenson, Sayre, *Chem. Abstracts*, 1916, **10**, 804.

#### Senecifoline

$C_{18}H_{22}O_8N$  MW, 385

Plates from  $CHCl_3$ -pet. ether. M.p. 194–5°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $H_2O$ , pet. ether.  $[\alpha]_D + 28.8^\circ$  in EtOH.

$B, HCl$ : m.p. 260° decomp.  $[\alpha]_D - 20^\circ$  in  $H_2O$ .

$B, HI$ : m.p. 248° decomp.

$B, HNO_3$ : m.p. 240° decomp.

$B, HAuCl_4$ : m.p. 220° decomp.

Watt, *J. Chem. Soc.*, 1909, **95**, 469.

#### Senecifolinene.

See Retronecine.

#### Senecioid Acid.

See 2 : 2-Dimethylacrylic Acid.

#### Seneciophylline

$C_{17}H_{23}O_5N$  MW, 321

Occurs in *Senecio platyphyllus*, D.C. Plates from EtOH. M.p. 217–18° decomp. Sol.  $CHCl_3$ . Mod. sol. EtOH,  $Me_2CO$ . Spar. sol.  $Et_2O$ , ligroin.  $[\alpha]_D - 128.04^\circ$  in  $CHCl_3$ .

Perchlorate : decomp. at 245°.

Aurichloride : m.p. 162–3° decomp.

Platinichloride : decomp. at 240°.

Picrate : m.p. 182–3°.

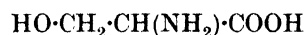
Methiodide : m.p. 231–2° decomp.

Orechoff, *Ber.*, 1935, **68**, 654.

#### Sennite.

See under Inositol.

**Serine** (1-Aminohydracrylic acid, 2-hydroxy-1-aminopropionic acid, 2-hydroxy- $\alpha$ -alanine)



$C_3H_7O_3N$

MW, 105

Widely distributed in animal proteins.

*d.*

Prisms from  $H_2O$ . M.p. 228° decomp.  $[\alpha]_D^{20} + 6.87^\circ$  in  $H_2O$ .

*l.*

$[\alpha]_D^{20} - 6.83^\circ$  in  $H_2O$ .

*Me ester* : hydrochloride, m.p. 167°.

*dl.*

Leaflets from  $H_2O$ . M.p. 246° decomp. Sol.  $H_2O$ . Insol. EtOH,  $Et_2O$ .  $H_2O_2 + FeSO_4 \rightarrow HO-CH_2-CHO$ .  $FeCl_3 \rightarrow$  red col.

*Me ester* :  $C_4H_9O_3N$ . MW, 119.  $B, HCl$ : m.p. 114°.

*N*-Chloroacetyl : m.p. 122–3°.

*N*-1-Bromopropionyl : m.p. about 143°.

*Benzoyl deriv.* : decomp. at 149–50°.  $B, HCl$ : m.p. 185–6°. *Picrate* : m.p. 168–9°.

*Phosphoric ester* : serine-phosphoric acid.  $C_3H_8O_6NP$ . Ba salt forms white flocks in hot  $H_2O$ .

*Et ether* :  $C_5H_{11}O_3N$ . MW, 133. Needles from EtOH. M.p. 256° decomp.

*Picrolonate* : decomp. at 265°.

*Phenylurethane* : m.p. 159°.

Leuchs, Geiger, *Ber.*, 1906, **39**, 2645.

Fischer, Jacobs, *ibid.*, 2944.

Mitra, *J. Indian Chem. Soc.*, 1930, **7**, 799.

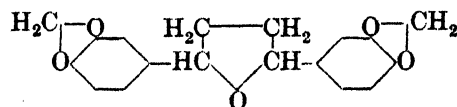
Dunn, Redemann, Smith, *J. Biol. Chem.*, 1934, **104**, 511 (*Bibl.*).

Levene, Schormüller, *J. Biol. Chem.*, 1934, **106**, 595; **105**, 547.

#### Serine-phosphoric Acid.

See under Serine.

#### Sesamin



$C_{18}H_{16}O_5$

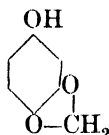
MW, 312

Occurs in sesame oil. Needles from EtOH. M.p. 123°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O, pet. ether.  $[\alpha]_D^{25} + 68.23^\circ$  in CHCl<sub>3</sub>.

Kreis, *Chem. Abstracts*, 1930, **24**, 2000.  
Adriani, *Chem. Abstracts*, 1929, **23**, 2054.  
Böesecken, Cohen, *Biochem. Z.*, 1928, **201**, 454.

Bertram, van der Steur, Waterman, *Biochem. Z.*, 1928, **197**, 1.

**Sesamol** (3:4-Methylenedioxyphenol, 1:2:4-trihydroxybenzene 1:2-methylene ether)



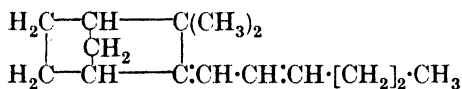
C<sub>7</sub>H<sub>6</sub>O<sub>3</sub> MW, 126

M.p. 65.8°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, pet. ether. FeCl<sub>3</sub> → violet-brown col. Colours rapidly in air.

β-Glucoside: C<sub>13</sub>H<sub>16</sub>O<sub>8</sub>. Cryst. + 1H<sub>2</sub>O. M.p. 168–9°.

Böesecken, Cohen, Kip, *Rec. trav. chim.*, 1936, **55**, 815.

### Sesquicamphene



C<sub>15</sub>H<sub>24</sub> MW, 204

Occurs in camphor oil. B.p. 255°, 140°/10 mm., 129–33°/8 mm. D<sub>20</sub> 0.9015. n<sub>D</sub> 1.50058.  $[\alpha]_D^{25} + 73.5^\circ$ .

Semmler, Rosenberg, *Ber.*, 1913, **46**, 768.  
Langlois, *Ann. chim.*, 1919, **12**, 358.

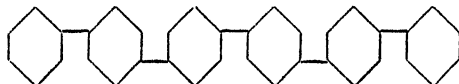
### Sesquic citronellene

C<sub>15</sub>H<sub>24</sub> MW, 204

Occurs in Java citronella oil. B.p. 138–40°/9 mm. D<sub>20</sub> 0.8489. n<sub>D</sub> 1.53252.

Semmler, Spornitz, *Ber.*, 1913, **46**, 4025.

### Sexiphenyl

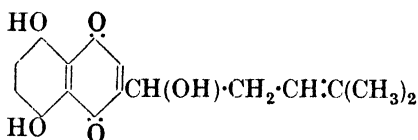


C<sub>36</sub>H<sub>26</sub> MW, 458

Cryst. from o-dichlorobenzene. M.p. 465°. Sol. quinoline, decahydronaphthalene.

Pummerer, *Ber.*, 1924, **57**, 84; 1933, **66**, 802.

### Shikonin (d-Alkannin)



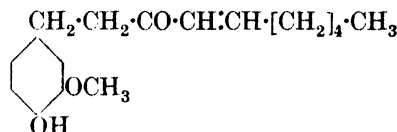
C<sub>16</sub>H<sub>16</sub>O<sub>5</sub> MW, 288

Occurs in root of *Lithospermum erythrorhizon*, ("shikon"). Brownish-red needles from C<sub>6</sub>H<sub>6</sub>. M.p. 143°. Reduces Tollen's. FeCl<sub>3</sub> → indigo-blue ppt.

Me ether: C<sub>17</sub>H<sub>18</sub>O<sub>5</sub>. MW, 302. M.p. 105°.

Karoda, Wada, *Chem. Zentr.*, 1937, **I**, 3156.

### Shogaol



C<sub>17</sub>H<sub>24</sub>O<sub>3</sub> MW, 276

Occurs in ginger ("shoga"). Pale yellow oil. B.p. 201.3°/2–2.5 mm. Sol. EtOH, AcOH. D<sub>20</sub> 1.0419. n<sub>D</sub> 1.52518. FeCl<sub>3</sub> → green col. Reduces Tollen's.

Acetyl deriv.: b.p. 183–8°/0.6 mm.

Me ether: C<sub>18</sub>H<sub>26</sub>O<sub>3</sub>. MW, 290. Yellow oil. B.p. 160–5°/0.06 mm.

Et ether: C<sub>19</sub>H<sub>28</sub>O<sub>3</sub>. MW, 304. Yellow oil. B.p. 181–6°/0.65 mm.

Nomura, Iwamoto, Murakami, *Chem. Abstracts*, 1930, **24**, 2445.

### Shonanic Acid

C<sub>10</sub>H<sub>14</sub>O<sub>2</sub> MW, 166

Occurs in wood of *Libocedrus formosana*, Florin. Cryst. from pet. ether. M.p. 40–1°. B.p. 264°/754 mm., 134–134.5°/6 mm. D<sub>4</sub><sup>20</sup> 1.016. n<sub>D</sub><sup>20</sup> 1.4842.  $[\alpha]_D^{25} - 0.75^\circ$  in EtOH.

Me ester: C<sub>11</sub>H<sub>16</sub>O<sub>2</sub>. MW, 180. B.p. 222°, 113–14°/20 mm. D<sub>4</sub><sup>20</sup> 0.9848. n<sub>D</sub><sup>20</sup> 1.4758.  $[\alpha]_D^{25} - 2.84^\circ$ .

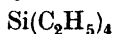
Et ester: C<sub>12</sub>H<sub>18</sub>O<sub>2</sub>. MW, 194. B.p. 228–9°/759 mm., 106–8°/7 mm. D<sub>4</sub><sup>20</sup> 0.9568. n<sub>D</sub><sup>20</sup> 1.4674.  $[\alpha]_D^{25} - 4.24^\circ$ .

Chloride: C<sub>10</sub>H<sub>13</sub>OCl. MW, 184.5. B.p. 215°, 106–7°/20 mm. D<sub>4</sub><sup>20</sup> 1.0577. n<sub>D</sub><sup>20</sup> 1.4955.

Amide: C<sub>10</sub>H<sub>15</sub>ON. MW, 165. M.p. 116–17°.

Anilide: m.p. 111–12°.

Ichikawa, *Bull. Chem. Soc. Japan*, 1936, **11**, 759.

**Silicon tetraethyl**

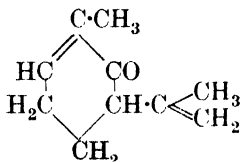
$\text{C}_8\text{H}_{20}\text{Si}$  MW, 144  
B.p. 152.8–153.2°/758.5 mm.  $D_4^{25}$  0.7620.  
 $n_D^{25}$  1.4246.

Kipping, Lloyd, *J. Chem. Soc.*, 1901, **79**, 456.

Bygden, *Ber.*, 1911, **44**, 2650.

**Silvan.**

See 2-Methylfuran.

**Silvecarvone** ( $\Delta^{6,8(9)}$ -*m*-Menthadienone-2)

$\text{C}_{10}\text{H}_{14}\text{O}$  MW, 150

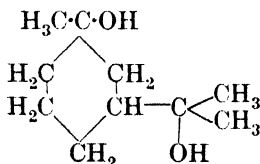
Oil.

Semicarbazone: m.p. 175–7°.

Wallach, *Ann.*, 1907, **357**, 74.

**Silver Salt.**

See under Anthraquinone-2-sulphonic Acid.

**Silveterpin** (d-*m*-Menthandiol-1 : 8)

$\text{C}_{10}\text{H}_{20}\text{O}_2$  MW, 172

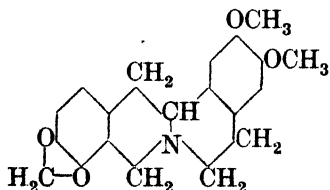
$\alpha$ -.

M.p. 137–8°.  $[\alpha]_D + 27.74^\circ$  in  $\text{CHCl}_3$ . Sublimes.

$\beta$ -.

M.p. 70–5°.  $[\alpha]_D + 20.93^\circ$  in  $\text{CHCl}_3$ .

Haworth, Perkin, Wallach, *J. Chem. Soc.*, 1913, **103**, 1233; *Ann.*, 1913, **399**, 161.

**Sinactine** (Tetrahydroepiberberine)

$\text{C}_{20}\text{H}_{21}\text{O}_4\text{N}$  MW, 339

*l*-.

Occurs in *Sinomenium acutum*. Prisms from EtOH. M.p. 175°. Sol.  $\text{CHCl}_3$ . Spar. sol.

MeOH, EtOH. Insol.  $\text{H}_2\text{O}$ .  $[\alpha]_D - 312^\circ$  in  $\text{CHCl}_3$ .

*B, HCl*: m.p. about 272° decomp.

*B, H, PtCl*: m.p. 245–7°.

*dl*-.

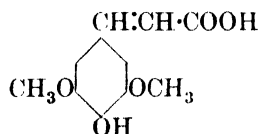
Needles from EtOH. M.p. 168°.

*B, HCl*: m.p. about 286° decomp.

Goto, Kitasato, *J. Chem. Soc.*, 1930, 1234.

**Sinamin.**

See Triallyltricyanamide.

**Sinapic Acid** (Sinapinic acid, 4-hydroxy-3 : 5-dimethoxycinnamic acid)

$\text{C}_{11}\text{H}_{12}\text{O}_5$  MW, 224

Yellow needles from EtOH. M.p. 192°. Sol. hot EtOH. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Ox.  $\rightarrow$  2 : 6-dimethoxy-*p*-benzoquinone.  $\text{FeCl}_3 \rightarrow$  red col.

Carbethoxyl: m.p. 174°.

Acetyl: m.p. 188–93°. Chloride: m.p. 142–4°.

Me ester:  $\text{C}_{12}\text{H}_{14}\text{O}_5$ . MW, 238. Cryst. from MeOH.Aq. M.p. 91–2°.

Et ester:  $\text{C}_{13}\text{H}_{16}\text{O}_5$ . MW, 252. Cryst. +  $\text{H}_2\text{O}$  from EtOH.Aq. M.p. 80–1°.

Choline ester: see Sinapin.

4-Me ether: 3 : 4 : 5-trimethoxycinnamic acid.

$\text{C}_{13}\text{H}_{14}\text{O}_5$ . MW, 238. Needles from  $\text{H}_2\text{O}$ . M.p. 123.5–124.5° (126.8°). Sol. hot  $\text{H}_2\text{O}$ . Me ester:

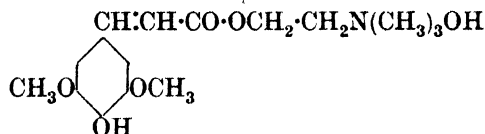
$\text{C}_{13}\text{H}_{16}\text{O}_5$ . MW, 252. Yellow leaflets from EtOH.Aq. M.p. 91–91.5°.

Späth, *Monatsh.*, 1920, **41**, 271.

Mauthner, *Ber.*, 1908, **41**, 2531.

Bogert, Isham, *J. Am. Chem. Soc.*, 1914, **36**, 519.

Chmielewska, *Chem. Zentr.*, 1937, **I**, 3156.

**Sinapin** (4-Hydroxy-3 : 5-dimethoxycinnamic acid choline ester)

$\text{C}_{16}\text{H}_{25}\text{O}_6\text{N}$  MW, 327

Occurs in black mustard seeds. Unstable.

Hyd.  $\rightarrow$  sinapic acid + choline.

Salts ( $B = \text{C}_{16}\text{H}_{23}\text{O}_5\text{N}$ ):—

*B, HBr*: m.p. anhyd. 107–15°.

*B,HI* : m.p. 185–6°.

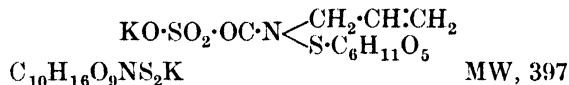
 $B, H_2SO_4$ : m.p. anhyd. 126.5–127.5°.

See first reference above.

**Sincalin.**

*See Choline.*

**Sinigrin** (*Sinigroside, potassium myronate*)



Occurs in black mustard seeds and *Alliaria officinalis*, D.C. M.p. 127-9°.  $[\alpha]_D^{18} - 16.4^\circ$  in  $H_2O$ .

Benik, Brauss, *Z. physiol. Chem.*, 1923,  
126, 210.

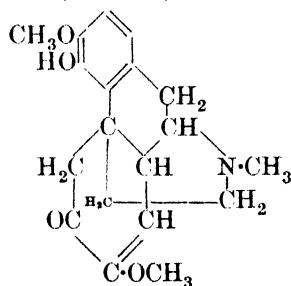
Herissey, Boivin, *Chem. Abstracts*, 1928, 22, 667.

Schneider, Fischer, Specht, *Ber.*, 1930,  
63, 2789.

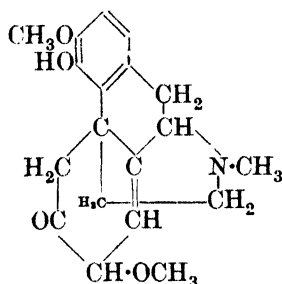
**Sinigroside.**

*See Sinigrin.*

### Sinomenine (Coculine)



**or**


$$\text{C}_{10}\text{H}_{22}\text{O}_4\text{N} \quad \text{MW, 329}$$

Occurs in *Sinomenium acutum*. Needles from  $C_6H_6$ . M.p.  $162^\circ$ . Sol. EtOH,  $CHCl_3$ ,  $Me_2CO$ , amyl alcohol. Mod. sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ . Insol. pet. ether.  $[\alpha]_D^{25} - 70-70^\circ$ .

*B, HCl*: m.p. 231°.

*B, HBr*: m.p. 231°.

*B,HI*: m.p. 233°.

$B, HNO_3$ : m.p.  $215^\circ$  decomp.

*Oxime* : m.p. 233°.

*Picrate* : m.p. about 140°.

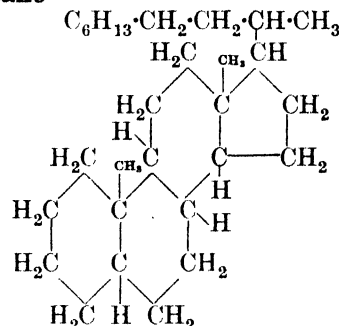
*Methiodide* : decomp. at 255°.

Goto, Michinaki, Shishido, *Ann.*, 1935,  
515, 297.

**Sinomenol.**

See under 2:3:5:6-Tetrahydroxyphenanthrene.

## Sitostane


$$\text{C}_{29}\text{H}_{52} \quad \text{MW, 400}$$

Derived from  $\beta$ -sitosterol. Cryst. from EtOH. M.p. 84.5–85°.  $[\alpha]_D^{20} + 26.9^\circ$  in  $\text{CHCl}_3$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

Windaus, Rahlen, *Z. physiol. Chem.*,  
1918, **101**, 223.

*Note.*—According to Bengtsson, *Z. physiol. Chem.*, 1935, **237**, 46, it is highly probable that sitostane is identical with stigmastane.

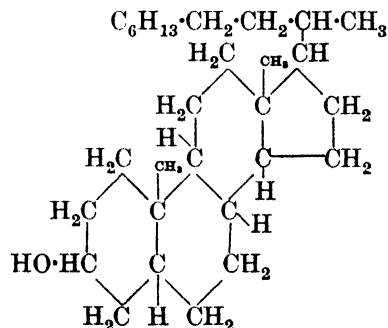
### $\gamma$ -Sitostane

$$\text{C}_{29}\text{H}_{52}(\text{C}_{28}\text{H}_{50}) \quad \text{MW, 400 (386)}$$

Derived from  $\gamma$ -sitosterol. M.p.  $87^{\circ}$ .  $[\alpha]_D^{15} + 20.2^{\circ}$  in  $\text{CHCl}_3$ .

Bonstedt, Z. *physiol. Chem.*, 1928, **176**, 279.

**Sitostanol** (*Dihydrositosterol, ostreastanol*)


$$\text{C}_{90}\text{H}_{59}\text{O} \quad \text{MW, 416}$$

Minor constituent of sitosterol complex.  
Derived from  $\beta$ -sitosterol. Plates from EtOH.

M.p. 139–139.5° (141°).  $[\alpha]_D^{20} + 25.6^\circ$  in  $\text{CHCl}_3$ . Sol. common org. solvents.

*Acetyl*: plates from  $\text{AcOEt}$ . M.p. 137° (132°).  $[\alpha]_D^{20} + 15.1^\circ$  in  $\text{CHCl}_3$ .

*Benzoyl*: plates from  $\text{AcOEt}$ . M.p. 137.6–138.6°.

3:5-Dinitrobenzoyl: plates from  $\text{AcOEt}$ . M.p. 214–15°.  $[\alpha]_D + 14.0^\circ$  in  $\text{C}_6\text{H}_6$ .

*Phenylurethane*: cryst. from  $\text{EtOH}$ . M.p. 175°.

Anderson, Shriner, Burr, *J. Am. Chem. Soc.*, 1926, **48**, 2987.

Bergmann, *J. Biol. Chem.*, 1934, **104**, 553.

*Note*.—For the possible identity of sitostanol with stigmastanol see

Bengtsson, *Z. physiol. Chem.*, 1935, **237**, 46.

### $\gamma$ -Sitostanol

$\text{C}_{29}\text{H}_{52}\text{O}$  ( $\text{C}_{28}\text{H}_{50}\text{O}$ ) MW, 416 (402)

Plates from  $\text{EtOH}$ . M.p. 143–4°.  $[\alpha]_D^{16.5} + 20.8^\circ$  in  $\text{CHCl}_3$ .

*Acetyl*: m.p. 144–5°.  $[\alpha]_D^{20} + 12.4^\circ$  in  $\text{CHCl}_3$ .

Bonstedt, *Z. physiol. Chem.*, 1928, **176**, 278.

### Sitosterol

The phytosterol originally termed sitosterol is a complex mixture separated only with difficulty. Hence the purity of the following fractions must be accepted with reserve.

$\alpha_1$ .

$\text{C}_{29}\text{H}_{48}\text{O}$  MW, 412

Widely distributed in plants in small amounts. Needles from  $\text{EtOH}$ . M.p. 164–6°.  $[\alpha]_D^{28} - 1.7^\circ$  in  $\text{CHCl}_3$ . Precipitated by digitonin.

*Acetyl*: plates from  $\text{EtOH}$ . M.p. 137°.  $[\alpha]_D^{28} + 28.6^\circ$  in  $\text{CHCl}_3$ .

*Benzoyl*: needles from  $\text{EtOH}-\text{C}_6\text{H}_6$ . M.p. 168–72°.  $[\alpha]_D^{27} + 41.8^\circ$  in  $\text{CHCl}_3$ .

3:5-Dinitrobenzoyl: plates from  $\text{AcOEt}$ . M.p. 222°.  $[\alpha]_D^{24} + 37.2^\circ$  in  $\text{CHCl}_3$ .

Wallis, Fernholz, *J. Am. Chem. Soc.*, 1936, **58**, 2446.

$\alpha_2$ .

$\text{C}_{30}\text{H}_{50}\text{O}$  MW, 426

Widely distributed in plants in small amounts. Cryst. from  $\text{EtOH}$ -pet. ether. M.p. 156°.  $[\alpha]_D^{25} + 3.5^\circ$  in  $\text{CHCl}_3$ . Sol. common org. solvents.

*Acetyl*: plates from  $\text{EtOH}$ . M.p. 124–6°.  $[\alpha]_D^{27} + 16.5^\circ$  in  $\text{CHCl}_3$ . Spar. sol.  $\text{MeOH}$ .

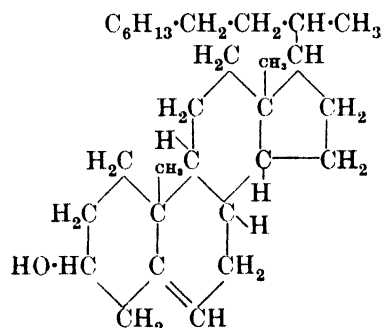
*Benzoyl*: needles from  $\text{EtOH}-\text{C}_6\text{H}_6$ . M.p.

164–6°.  $[\alpha]_D^{28} + 27.4^\circ$  in  $\text{CHCl}_3$ . Spar. sol.  $\text{EtOH}$ .

3:5-Dinitrobenzoyl: needles from  $\text{Me}_2\text{CO}$ . M.p. 206°.  $[\alpha]_D^{20} + 26.4^\circ$  in  $\text{CHCl}_3$ .

See previous reference.

$\beta$ -.



$\text{C}_{29}\text{H}_{50}\text{O}$  MW, 414

Widely distributed in plants. Needles from  $\text{MeOH}$ . M.p. 136–7°.  $[\alpha]_D^{22} - 35^\circ$  in  $\text{CHCl}_3$ .

*Acetyl*: needles from  $\text{MeOH}$ . M.p. 134° (128°).  $[\alpha]_D^{22} - 38.5^\circ$  in  $\text{CHCl}_3$ .

*Benzoyl*: m.p. 145.5°.

3:5-Dinitrobenzoyl: m.p. 203°.  $[\alpha]_D^{22} - 10.6^\circ$  in  $\text{CHCl}_3$ .

Anderson, Shriner, Burr, *J. Am. Chem. Soc.*, 1926, **48**, 2987.

Ichiba, *Chem. Zentr.*, 1936, I, 1027.

$\gamma$ -.

$\text{C}_{29}\text{H}_{50}\text{O}$  ( $\text{C}_{28}\text{H}_{48}\text{O}$ ) MW, 414 (400)

Plates from  $\text{EtOH}$ . M.p. 147–8°.  $[\alpha]_D - 43.13^\circ$  in  $\text{CHCl}_3$ .

*Acetyl*: cryst. from  $\text{EtOH}$ . M.p. 143°.  $[\alpha]_D - 47.7^\circ$  in  $\text{CHCl}_3$ . *Dibromide*: needles from  $\text{C}_6\text{H}_6-\text{MeOH}$ . M.p. 140–1°.  $[\alpha]_D - 46.23^\circ$  in  $\text{CHCl}_3$ .

*Benzoyl*: m.p. 152°.  $[\alpha]_D - 19.63^\circ$  in  $\text{CHCl}_3$ .

Ichiba, *Chem. Zentr.*, 1936, I, 1027.

Sandqvist, Bengtsson, *Ber.*, 1931, **64**, 2167.

$\delta$ -.

$\text{C}_{29}\text{H}_{50}\text{O}$  MW, 414

Cryst. from  $\text{EtOH}$ . M.p. 146–7°.  $[\alpha]_D - 23.9^\circ$  in  $\text{CHCl}_3$ .

*Acetyl*: m.p. 115°.  $[\alpha]_D - 24.35^\circ$  in  $\text{CHCl}_3$ .

*Benzoyl*: m.p. 157–8°.  $[\alpha]_D - 15.98^\circ$  in  $\text{CHCl}_3$ .

See first reference above.

$\epsilon$ -.

$\text{C}_{29}\text{H}_{50}\text{O}$  MW, 414

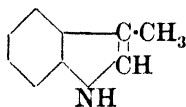
M.p. 143–4°.  $[\alpha]_D^{17} - 38.7^\circ$  in  $\text{CHCl}_3$ .

*Acetyl*: plates from EtOH. M.p. 127–8°.  $[\alpha]_D^{17} - 44.7^\circ$  in  $\text{CHCl}_3$ .

*3:5-Dinitrobenzoyl*: m.p. 215–17°.  $[\alpha]_D^{17} - 10^\circ$  in  $\text{CHCl}_3$ .

Simpson, *J. Chem. Soc.*, 1927, 737.

**Skatole** (*Scatole*, 3-methylindole,  $\beta$ -methylindole)



$\text{C}_9\text{H}_9\text{N}$

MW, 131

Occurs in coal tar, beetroot, nectandra wood and faeces. Leaflets from ligroin. M.p. 95°. B.p. 265–6°/755 mm.  $\text{K}_4\text{Fe}(\text{CN})_6 + \text{H}_2\text{SO}_4 \rightarrow$  violet col.

*B<sub>2</sub>HCl*: m.p. 167–8°.

*N-Acetyl*: m.p. 68°.

*N-Propionyl*: m.p. 45°.

*Picrate*: m.p. 170–1° decomp.

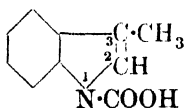
*N-Me*: see 1:3-Dimethylindole.

Kruber, D.R.P., 515,543, (*Chem. Abstracts*, 1931, 25, 2443).

King, l'Ecuyer, *J. Chem. Soc.*, 1934, 1903.

Fischer, *Ann.*, 1886, 236, 138.

**Skatole-1-carboxylic Acid** (3-Methylindole-N-carboxylic acid)



$\text{C}_{10}\text{H}_9\text{O}_2\text{N}$

MW, 175

Occurs in beet. Needles. M.p. 162.5°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Lippmann, *Ber.*, 1924, 57, 257.

**Skatole-2-carboxylic Acid** (3-Methylindole-2-carboxylic acid).

Needles from AcOH. M.p. 164–5° (167°). Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, ligroin.  $k = 4.7 \times 10^{-6}$  at 25°.

*Et ester*: C<sub>12</sub>H<sub>13</sub>O<sub>2</sub>N. MW, 203. Needles from EtOH. M.p. 133–4°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Amide*: C<sub>10</sub>H<sub>10</sub>ON<sub>2</sub>. MW, 174. Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 115°.

*N-Me*: C<sub>11</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 189. Cryst. from C<sub>6</sub>H<sub>6</sub>-EtOH. Decomp. about 213°. Sol. EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, pet. ether.

Perkin, Kermack, Robinson, *J. Chem. Soc.*, 1921, 119, 1634.

Oddo, *Gazz. chim. ital.*, 1912, 42, i, 370.

**Smilagenin**

$\text{C}_{27}\text{H}_{44}\text{O}_3$

MW, 416

Occurs in Jamaica sarsaparilla root. Needles from Me<sub>2</sub>CO. M.p. 183–4°.  $[\alpha]_D^{25} - 69^\circ$  in  $\text{CHCl}_3$ .

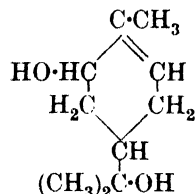
*Acetyl deriv.*: m.p. 150–1°.  $[\alpha]_D^{25} - 59.6^\circ$ .

*Benzoyl deriv.*: m.p. 181–181.5°.

Farmer, Kon, *J. Chem. Soc.*, 1937, 414.

Askew, Farmer, Kon, *J. Chem. Soc.*, 1936, 1402.

**Sobrerol** ( $\Delta^1$ -p-Menthenediol-6:8, 1-methyl-4- $\alpha$ -hydroxyisopropylcyclohexenol-6)



$\text{C}_{10}\text{H}_{18}\text{O}_2$

MW, 170

*d.*

Prisms from H<sub>2</sub>O, plates from EtOH. M.p. 150° (148–9°).  $[\alpha]_D + 141^\circ 16'$  in EtOH.

*l.*

Yellow cryst. M.p. 147–8°.

Dupont, *Chem. Abstracts*, 1922, 16, 4340.

Gildemeister, Köhler, *Chem. Zentr.*, 1909, II, 2159.

Prilezhaev, Vershuk, *Chem. Abstracts*, 1930, 24, 697.

Wallach, *Ann.*, 1917, 414, 196.

**Solaniidine-S**

$\text{C}_{18}\text{H}_{31}\text{ON}$

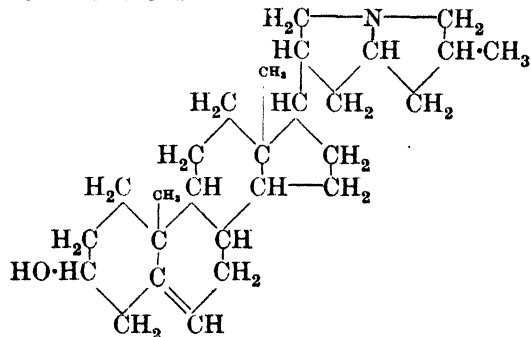
MW, 277

Occurs in *Solanum sodomæum*, Linn. Needles. M.p. 249–53°.

Rochelmeyer, *Chem. Zentr.*, 1937, I, 1947.

Oddo, Caronna, *Ber.*, 1936, 69, 283.

**Solaniidine-T**



$\text{C}_{27}\text{H}_{43}\text{ON}$

MW, 397



Occurs in *Solanum tuberosum*, Linn. Cryst. from EtOH. M.p. 219° (216°).

*Acetyl deriv.*: m.p. 204°.

Clemons, Morgan, Raper, *J. Chem. Soc.*, 1936, 1299.

Soltys, Wallenfels, *Ber.*, 1936, **69**, 811.

Heiduschka, Philippi, *Ber.*, 1935, **68**, 669.

Oddo, Caronna, *Ber.*, 1934, **67**, 451.

Dieterle, Rochelmeyer, *Arch. Pharm.*, 1935, **273**, 532.

### Solanocarpidine

$C_{26}H_{43}O_3N$  MW, 417

Plates from EtOH. M.p. 197–8°. Sol. ord. org. solvents.

*B.HCl*: m.p. 313–14° decomp.

*B.HBr*: m.p. 307–8° decomp.

*B.HI*: m.p. 283–4° decomp.

*B.H<sub>2</sub>SO<sub>4</sub>*: m.p. 293–4° decomp.

*B.HNO<sub>3</sub>*: m.p. 271–2° decomp.

*B.(COOH)<sub>2</sub>*: m.p. 238–9° decomp.

*Picrate*: m.p. 148–9°.

Saiyed, Kanga, *Chem. Zentr.*, 1937, I, 2181.

### Solanocarpine

$C_{44}H_{77}O_{19}N$  MW, 923

Occurs in fruit of *Solanum xanthocarpum*, Schrad & Wendl. Needles from EtOH. M.p. 288–9° decomp. Sol. H<sub>2</sub>O. Hyd. → solanocarpidine + glucose + rhamnose + galactose (?)

See previous reference.

### Soneryl.

See Neonal.

### Sophocarpidine.

*Matrine, q.v.*

### Sophocarpine

$C_{15}H_{24}ON_2$  MW, 248

Cryst. + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 81–2°, anhyd. 54–5°.  $[\alpha]_D^{25}$  –29.44° in EtOH.

*Aurichloride*: m.p. 166–7°.

*Platinochloride*: m.p. 209–12° decomp.

*Methiodide*: needles from EtOH. M.p. 200–2°.

*Picrate*: m.p. 155–7°.

Orechhoff, Proskurnina, *Ber.*, 1934, **67**, 77.

### Sophoramine

$C_{15}H_{20}ON_2$  MW, 244

M.p. 164–5°.  $[\alpha]_D$  –90.85°.

*B.HCl*: m.p. 247–8° decomp.

*B.HI*: m.p. 294–6°.

*Aurichloride*: m.p. 183–4° decomp.

*Platinochloride*: m.p. 245–7° decomp.

*Picrate*: m.p. 229–31° decomp.

*Picrolonate*: m.p. 173–5° decomp.

Orechhoff, *Chem. Zentr.*, 1935, II, 2215.

Orechhoff, Proskurnina, Konowalowa, *Ber.*, 1935, **68**, 431.

### Sophoretin.

See Quercetin.

### Sophoricol.

*Genistein, q.v.*

### Sophoridine

$C_{15}H_{26}ON_2$  MW, 250

Needles from pet. ether. M.p. 109–10°.  $[\alpha]_D$  –63.57°.

*Aurichloride*: m.p. 189–90°.

*Methiodide*: m.p. 234–6°.

*Picrolonate*: m.p. 226–8° decomp.

Orechhoff, *Chem. Zentr.*, 1935, II, 2215.

Orechhoff, Proskurnina, Konowalowa, *Ber.*, 1935, **68**, 431.

### Sophorine.

See Cytisine.

**Sorbic Acid** (1:3-Hexadienic acid, 1:3-pentadiene-1-carboxylic acid, 2-propenylacrylic acid)



$C_6H_8O_2$  MW, 112

Needles from EtOH.Aq. M.p. 132–133.5° (133–4°). B.p. 228° decomp. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. hot H<sub>2</sub>O. Volatile in steam. Heat of comb. *C<sub>p</sub>* 743.4 Cal., *C<sub>e</sub>* 742.8 Cal. *k* = 1.73 × 10<sup>–5</sup> at 25°.

*Me ester*:  $C_7H_{10}O_2$ . MW, 126. Leaflets. M.p. 5°. B.p. 180°/759 mm. (174°), 70°/20 mm.

*Et ester*:  $C_8H_{12}O_2$ . MW, 140. B.p. 195.5°, 85°/20 mm., 76.5°/12 mm. *D*<sub>4</sub><sup>20</sup> 0.9560. *n*<sub>D</sub><sup>20</sup> 1.502.

*Chloride*:  $C_6H_7OCl$ . MW, 130.5. B.p. 78°/15 mm.

*Amide*:  $C_6H_9ON$ . MW, 111. Needles from H<sub>2</sub>O. M.p. 168°. Sol. H<sub>2</sub>O, EtOH.

*Nitrile*:  $C_6H_7N$ . MW, 93. B.p. 72°/20 mm., 50–60°/12 mm.

*Anilide*: m.p. 153°.

*o-Toluidide*: m.p. 173°.

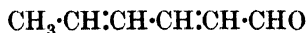
*Phenylhydrazide*: m.p. 162–3°.

Philippi, *Monatsh.*, 1929, **51**, 278.

Doebner, Wolff, *Ber.*, 1901, **34**, 2221.

Baumgarten, Glatzel, *Ber.*, 1926, **59**, 2663.

**Sorbic Aldehyde** (1:3-Hexadienal, sorb-aldehyde)



$\text{C}_6\text{H}_8\text{O}$  MW, 96

B.p. 173–4°/754 mm., 76°/30 mm., 64–6°/11 mm.  $D_4^{20}$  0.9087.  $n_D^{20}$  1.5372. Reduces Tollen's rapidly, cold Fehling's slowly.

Oxime: needles from EtOH. M.p. 159–5–160.5° decomp.

Semicarbazone: plates from EtOH. M.p. 206°.

Phenylhydrazone: yellow plates from EtOH. M.p. 101–2°.

Fischer, Wiedemann, *Ann.*, 1934, **513**, 256.

Reichstein, Ammann, Trivelli, *Helv. Chim. Acta*, 1932, **15**, 261.

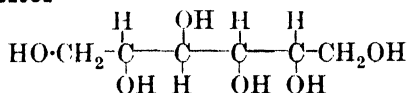
Kuhn, Hoffer, *Ber.*, 1930, **63**, 2164; 1931, **64**, 1978.

Baumgarten, Glatzel, *Ber.*, 1926, **59**, 2662.

#### Sorbierite.

See Iditol.

#### Sorbitol



$\text{C}_6\text{H}_{14}\text{O}_6$  MW, 182

*d.*

Occurs in ripe mountain ash berries, cherries, plums, pears, apples, medlars, fruit of cherry laurel, etc. Needles +  $\frac{1}{2}$  or  $1\text{H}_2\text{O}$ . M.p. 110–11° (anhyd.), 75° (hydrate), 87–95° from EtOH. Sweet taste. Sol.  $\text{H}_2\text{O}$ . Spar. sol. cold EtOH.  $[\alpha]_D^{15} - 1.73^\circ$  in  $\text{H}_2\text{O}$ ,  $[\alpha]_D^{20} - 2.01^\circ$  in  $\text{H}_2\text{O}$ .  $\text{HI} \rightarrow \text{sec.-n-hexyl iodide}$ . Sorbose bacterium  $\rightarrow l\text{-sorbse}$ .

Hexa-acetyl: cryst. from EtOH. M.p. 99.5° (120°).  $[\alpha]_D^{15} + 6.8^\circ$  in  $\text{Me}_2\text{CO}$ .

Dibenzoyl deriv.: m.p. 140°.  $[\alpha]_D^{15} + 1.69 - 1.85^\circ$  in Py.

Pentabenzoyl deriv.: m.p. 222°.  $[\alpha]_D^{15} + 24.54^\circ$  in Py.

Hexabenzoyl: m.p. 129°.  $[\alpha]_D^{15} + 24.3^\circ$  in Py.

Tetra-Me ether: b.p. 125°/0.4 mm.  $n_D$  1.4568.

$[\alpha]_D - 6.2^\circ$  in  $\text{H}_2\text{O}$ .

Penta-Et ether: b.p. 185–90°/10 mm.

Triacetone deriv.: m.p. 46–7°.  $[\alpha]_D^{15} + 12.7^\circ$

in EtOH.

Benzylidene deriv.: cryst. M.p. 175°.  $[\alpha]_D + 6^\circ$  in EtOH.

Dibenzylidene deriv.: cryst. M.p. 163°.

$[\alpha]_D + 29^\circ$  in  $\text{Me}_2\text{CO}$ .

*o*-Chlorobenzylidene deriv.: m.p. 170°.

Tri-*o*-chlorobenzylidene deriv.: m.p. 217°.

2:6-Dichlorobenzylidene deriv.: m.p. 204.5°.

2-Nitro-5-chlorobenzylidene deriv.: m.p. 250.5°.

*m*-Nitrobenzylidene deriv.: m.p. 180°.

Di-*m*-Nitrobenzylidene deriv.: m.p. 228.5°.

Tri-*o*-nitrobenzylidene deriv.: m.p. 181° (two forms, m.p. 212–15° and 142–6°).

Tri-*m*-nitrobenzylidene deriv.: m.p. 168°.

*l.*

Needles +  $\frac{1}{2}\text{H}_2\text{O}$ . M.p. 77°.

Dibenzylidene deriv.: cryst. M.p. 160°.

$[\alpha]_D - 28^\circ$ .

Boussingault, *Ann. chim. phys.*, 1872, **26**, 376.

Meunier, *Compt. rend.*, 1890, **111**, 49.

Vincent, Delachanal, *ibid.*, 51.

Fischer, *Ber.*, 1890, **23**, 3684.

Fischer, Stahel, *Ber.*, 1891, **24**, 2144.

Lobry de Bruyn, v. Ekenstein, *Rec. trav. chim.*, 1899, **18**, 151; 1900, **19**, 7.

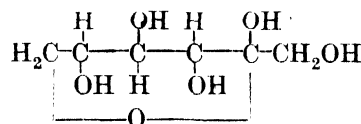
Asahina, Shinoda, *J. Pharm. Soc. Japan*, 1930, **50**, 1.

Davis, Slater, Smith, *Biochem. J.*, 1926, **20**, 1155.

Böeseken, Leevers, *Rec. trav. chim.*, 1935, **54**, 861.

Bleyer, Diemair, Lix, *Chem. Abstracts*, 1933, **27**, 4624.

#### Sorbse



$\text{C}_6\text{H}_{12}\text{O}_6$  MW, 180

*d.*

Cryst. M.p. 165°. Sweet taste. Sol.  $\text{H}_2\text{O}$ . Spar. sol. MeOH, EtOH.  $[\alpha]_D^{20} + 42.9^\circ$  in  $\text{H}_2\text{O}$ .  $D^{15}$  1.654. Reduces Fehling's. Non-fermentable.

Phenylosazone: m.p. 168° (156°, 160°). Identical with *d*-gulosazone and *d*-idosazone.

$\beta$ -Methylglucoside: cryst. M.p. 119°.  $[\alpha]_D + 88.5^\circ$  in  $\text{H}_2\text{O}$ .

*l.*

Cryst. M.p. 165° (162°, 159–61°). Sweet taste. Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH.  $[\alpha]_D^{20} - 42.9^\circ$  in  $\text{H}_2\text{O}$  (–43.2° in  $\text{H}_2\text{O}$ ).  $D^{15}$  1.654. Reduces Fehling's. Heat of comb.  $\text{C}_v$  3714.5 Cal.  $\text{NaHg} \rightarrow \text{sorbitol} + \text{iditol}$ . Non-fermentable.

Phenylosazone: yellow needles. M.p. 168° (164, 156°). Identical with *l*-gulosazone and *l*-idosazone. *p*-Bromophenylosazone: yellow needles. M.p. 181°.

*o*-Nitrophenylosazone : red powder. M.p. 211–12°.

$\beta$ -Methylglucoside : plates from Me<sub>2</sub>CO. M.p. 120–2°.  $[\alpha]_D^{20} = -88.5^\circ$  in H<sub>2</sub>O.

Penta-acetyl : m.p. 96.5–97.5°.  $[\alpha]_D + 2.9^\circ$  in CHCl<sub>3</sub>.

*dl.*  $\beta$ -Acrose.

Laminae. M.p. 162–3° (154°). D<sup>17</sup> 1.638 (1.634). Non-fermentable.

Phenylosazone : laminae from dil. EtOH. M.p. 169–70° decomp.

Schmitz, *Ber.*, 1913, **46**, 2334.

v. Ekenstein, Blanksma, *Rec. trav. chim.*, 1908, **27**, 1.

Schlubach, Vorwerk, *Ber.*, 1933, **66**, 1251.

Maurer, Schiedt, *Biochem. Z.*, 1934, **271**, 61.

Reichstein, Grussner, *Helv. Chim. Acta.*, 1934, **17**, 318.

Talen, *Rec. trav. chim.*, 1925, **44**, 891.

Böeseken, Leefers, *Rec. trav. chim.*, 1935, **54**, 861.

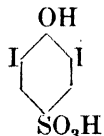
Fischer, Baer, *Helv. Chim. Acta*, 1936, **19**, 519.

Bernhauer, Görlich, *Biochem. Z.*, 1935, **280**, 375.

### Sorbyl Alcohol.

See 2 : 4-Hexadienol-1.

**Sozoiodolic Acid** (2 : 6-Di-iodophenol-*p*-sulphonic acid)



C<sub>6</sub>H<sub>4</sub>O<sub>4</sub>I<sub>2</sub>S MW, 426

Prisms + 3H<sub>2</sub>O. M.p. 120–120.5°. Sol. H<sub>2</sub>O. CrO<sub>3</sub> + H<sub>2</sub>SO<sub>4</sub> → 2 : 6-di-iodo-*p*-benzoquinone.

*Et* ether : C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>I<sub>2</sub>S. MW, 454. Cryst. + 2H<sub>2</sub>O. M.p. 108°. Sol. H<sub>2</sub>O.

*d*-Arginine salt : decomp. at 213–14°.

*Cadaverine* salt : decomp. at 242°.

*Choline* salt : decomp. at 180°.

*Creatinine* salt : decomp. at 229–31°.

*Glucosamine* salt : decomp. at 181–2°.

*Guanidine* salt : decomp. at 247–9°.

*Histamine* salt : decomp. at 241°.

*l*-Histidine salt : decomp. at 207–8°.

*Lysine* salt : decomp. at 234–5°.

*Putrescine* salt : decomp. at 250°.

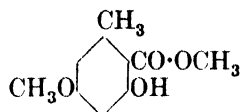
*Urea* salt : decomp. at 208°.

Tromsdorff, D.R.P., 45,226.

Rupp, Herrmann, *Arch. Pharm.*, 1916, **254**, 509.

Ackermann, *Z. physiol. Chem.*, 1934, **225**, 46.

**Sparassol** (*Methyl everninate*, 3-hydroxy-5-methoxy-*o*-toluic acid methyl ester)



C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>

MW, 196

Occurs in *Evernia prunastri*, Ach., *Sparassis ramosa*, Schäff., and *Rhododendron japonicum*, C. J. Schneider. M.p. 67–8°. FeCl<sub>3</sub> → violet col. in EtOH.

*Acetyl* : m.p. 41–2°.

*3-Me ether* : C<sub>11</sub>H<sub>14</sub>O<sub>4</sub>. MW, 210. M.p. 41–2°.

Kinoshita, *Chem. Abstracts*, 1931, **25**, 1552.

Wedekind, Fleischer, *Ber.*, 1924, **57**, 1121.

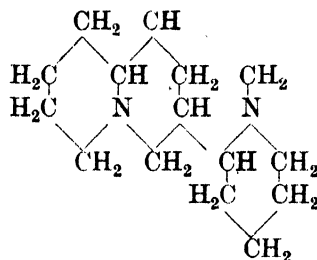
Späth, Jesekki, *ibid.*, 471.

Pfau, *ibid.*, 468.

Falck, *Ber.*, 1923, **56**, 2555.

Koller, Hamburg, *Monatsh.*, 1935, **65**, 375.

**Sparteine** (*Lupinidine*, *hexahydrodeoxy-anagyrine*, *pachycarpine*)



Probable structure

C<sub>15</sub>H<sub>26</sub>N<sub>2</sub>

MW, 234

*d.*

Occurs in seeds of *Anagyris foetida*, Linn., *Sophora pachycarpa*, Schrenk, *Thermopsis lanceolata*, R.Br. B.p. 173–4°/8 mm., 138°/2 mm., 133–5°/1 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. D<sub>4</sub><sup>20</sup> 1.027. n<sub>D</sub><sup>20</sup> 1.5312.  $[\alpha]_D^{20} + 16.1^\circ$  (+ 5.54°). Volatile in steam. Resinifies in air.

*B,2HCl* : m.p. 255–7°.

*B,HI* : m.p. 232–3°.

*B,HClO<sub>4</sub>* : plates from EtOH. M.p. 171–2°.

*B,H<sub>2</sub>PtCl<sub>6</sub>* : m.p. 240–1° decomp.

*Chloroaurate* : m.p. 192–3°.

*Dipicrate* : yellow needles from EtOH–Me<sub>2</sub>CO. M.p. 205–6°.

*Methiodide* : m.p. 236–8°.  $[\alpha]_D + 24.5^\circ$  in H<sub>2</sub>O.

l-.

B.p. 130–5°/1 mm.  $[\alpha]_D - 11.3^\circ$  in EtOH.*B,HI*: prisms from  $H_2O$ . M.p. 231°.*Dipicrate*: m.p. 205–6°.

dl-.

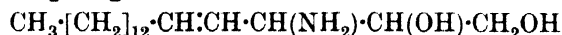
*Monopicrate*: m.p. 134–5°.*Dipicrate*: m.p. 205–6°.Mills, *Ann.*, 1863, 125, 71.Clemo, Leitch, Raper, *Ber.*, 1931, 64, 1520.Clemo, Raper, *J. Chem. Soc.*, 1933, 644.Clemo, Raper, Tenniswood, *J. Chem. Soc.*, 1931, 429.Orechhoff, Proskurnina, *Ber.*, 1935, 68, 1807.**Spermidine** ( $\omega$ -Aminopropyl- $\omega$ -aminobutylamine) $C_7H_{19}N_3$ 

MW, 145

Occurs in ox pancreas as phosphate. B.p. 128–30°/14 mm.

 $B_2(H_3PO_4)_3 \cdot 6H_2O$ : plates from EtOH.Aq. M.p. 207–8°. Sol.  $H_2O$ . $B_3HAuCl_4$ : needles from 1% HCl. M.p. 222°.*Tri-m-nitrobenzoyl*: m.p. anhyd. 148–50°.*Tripicrate*: yellow needles from  $H_2O$ . M.p. 211°.Dudley, Rosenheim, Starling, *Biochem. J.*, 1927, 21, 97.v. Braun, Pinkernelle, *Ber.*, 1937, 70, 1233.**Spermine** (*Gerontine, musculamine, neuridine, di-[\omega-aminopropyl]-tetramethylenediamine*) $C_{10}H_{26}N_4$ 

MW, 202

Occurs as phosphate in semen, ox pancreas, yeast, etc. Deliquescent cryst. Absorbs  $CO_2$  from the air.*Phosphate*: m.p. 230–4°. Sol. hot  $H_2O$ . Insol. EtOH,  $Et_2O$ .*Tetrabenzoyl*: needles from  $Me_2CO$ .Aq. M.p. 155°. $B_4HCl$ : needles. M.p. 310° decomp. $B_4HAuCl_4$ : golden leaflets from 5% HCl. M.p. 226° decomp. $B_2H_2PtCl_6$ : m.p. 242–5°.*Tetrapicrate*: yellow needles from  $H_2O$ . M.p. 248–50°.*Picrolonate*: pale yellow needles from  $H_2O$ . M.p. 288–9° decomp.Wrede, Fanselow, Strack, *Z. physiol. Chem.*, 1927, 163, 219.Dudley, Rosenheim, Rosenheim, *Biochem. J.*, 1924, 18, 1263.Wrede, Strack, Hettche, *Z. physiol. Chem.*, 1928, 173, 61.Dudley, Rosenheim, Starling, *Biochem. J.*, 1926, 20, 1082.**Sphingosine** $C_{18}H_{37}O_2N$ 

MW, 299

Needles from  $Et_2O$ . $B_2 \cdot H_2SO_4$ : m.p. 233–4° decomp.*Triacetyl deriv.*: m.p. 102–3°.  $[\alpha]_D^{19} - 24.09^\circ$ .*Lignoceryl deriv.*: m.p. 90–90.5°.*Picrolonate*: m.p. 87–9°.*Me ether*: *B,HCl*, plates from  $Me_2CO$ -EtOH. M.p. 141°.*Di-Me ether*: *B,HCl*, plates from  $Me_2CO$ -EtOH. M.p. 139° (133–4°).*Di-Et ether*: *B,HCl*, plates from  $Me_2CO$ . M.p. 113–15°.*Galactoside*: psychosine. Cryst. *Sulphate*: m.p. 225° decomp.  $[\alpha]_D - 16.6^\circ$ .Levene, Jacobs, *J. Biol. Chem.*, 1912, 11, 547.Klenk, Diebold, *Z. physiol. Chem.*, 1931, 198, 25.**Spinacene.**

See Squalene.

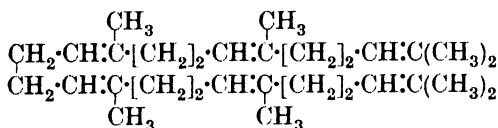
**Spinasterol** $C_{28}H_{46}O$ 

MW, 398

 $\alpha$ -.Occurs in spinach and senega root. Plates from EtOH. M.p. 172–5° (169–70°).  $[\alpha]_D - 3.7^\circ$  in  $CHCl_3$ .*Acetyl*: plates from AcOH. M.p. 187° (183–5°).  $[\alpha]_D^{23} - 4.7^\circ$  in  $CHCl_3$ .*Propionyl*: plates from AcOEt. M.p. 152–3°.  $[\alpha]_D^{23} - 5.0^\circ$  in  $CHCl_3$ .*Benzoyl*: plates from AcOEt. M.p. 201–2°.  $[\alpha]_D^{23} + 2.25^\circ$  in  $CHCl_3$ .*p-Nitrobenzoyl*: needles from AcOEt. M.p. 217–18°.  $[\alpha]_D^{23} + 4.5^\circ$  in  $CHCl_3$ .*Phenylurethane*: plates from AcOEt. M.p. 177° (173–4°).  $[\alpha]_D^{23} - 2.25^\circ$  in  $CHCl_3$ . $\beta$ -.Occurs in spinach. Cryst. from EtOH. M.p. 145–8°.  $[\alpha]_{5461} + 7.65^\circ$ .*Acetyl*: cryst. from AcOH. M.p. 150–4°.  $[\alpha]_{5461} + 7.2^\circ$ .

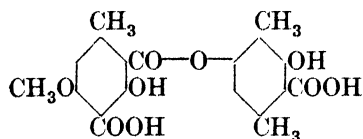
$\gamma$ -

Occurs in spinach. M.p. 159–60°.

*Acetyl*: m.p. 139.5–140°.  $[\alpha]_{D461} - 14.1^\circ$  in  $\text{CHCl}_3$ .*Benzoyl*: m.p. 118.5–119°.  $[\alpha]_{D461} - 10.3^\circ$  in  $\text{CHCl}_3$ .*p*-Nitrobenzoyl: m.p. 200°.  $[\alpha]_{D461} - 8.9^\circ$  in  $\text{CHCl}_3$ .*Phenylurethane*: m.p. 144–5°.  $[\alpha]_{D461} - 15.9^\circ$  in  $\text{CHCl}_3$ .Simpson, *J. Chem. Soc.*, 1937, 730.Heyl, Larsen, *J. Am. Chem. Soc.*, 1934, 56, 942, 2664; *Chem. Zentr.*, 1933, II, 2019.Hart, Heyl, *J. Biol. Chem.*, 1932, 95, 311.**Squalene** (*Spinacene*) $\text{C}_{30}\text{H}_{50}$ 

MW, 410

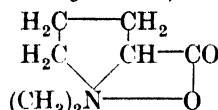
Occurs in oil of *Centrophorus granulosus*, Müller & Henle, *Scymnorhinus lichio*, *Etmopterus spinan*, *Lepidorhinus squamosus*. F.p. below  $-20^\circ$ . B.p. 284–5°/25 mm., 280°/17 mm., 260–2°/9 mm., 240–2°/4 mm., 105°/0.17 mm.  $D_{20}^{20} 0.8592$ .  $n_D^{20} 1.4990$ .

Challenger, *Industrial Chemist*, 1928, 4, 315 (*Bibl.*).Karrer, Helfenstein, *Helv. Chim. Acta*, 1931, 14, 78.Heilbron, Kamm, Owens, *J. Chem. Soc.*, 1926, 1630.**Squamatic Acid** $\text{C}_{19}\text{H}_{18}\text{O}_9$ 

MW, 390

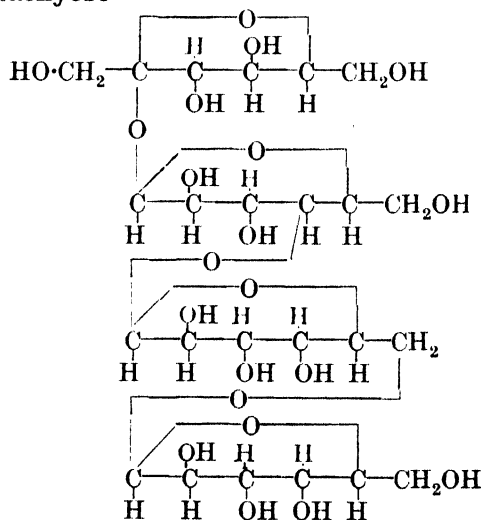
Occurs in the lichen *Cladonia bellidiflora*. Prisms from AcOH. M.p. 219°. Sol. AcOH, AcOEt. Spar. sol. EtOH, Et<sub>2</sub>O,  $\text{CHCl}_3$ .

*Di-Me ester*:  $\text{C}_{21}\text{H}_{22}\text{O}_9$ . MW, 418. M.p. 178°. *Di-Me ether*:  $\text{C}_{23}\text{H}_{26}\text{O}_9$ . MW, 446. Leaflets from MeOH. M.p. 135°.

Asahina, Tanase, *Ber.*, 1937, 70, 62.Asahina, Sakurai, *ibid.*, 64.**Stachydrine** (*Hygric acid methylbetaine*, *N-methylproline methylbetaine*) $\text{C}_7\text{H}_{13}\text{O}_2\text{N}$ 

MW, 143

Occurs in *Stachys tubrifera*, Naudin, and in fruit of *Citrus grandis*, Hassk, etc. Cryst. +  $\text{H}_2\text{O}$ . M.p. anhyd. 235° decomp. Sol.  $\text{H}_2\text{O}$ , EtOH. Insol. Et<sub>2</sub>O,  $\text{CHCl}_3$ . Decomp. in air.

*B, HCl*: cryst. from EtOH. M.p. 235° (211–15°).*B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: plates from 50% EtOH. Decomp. at 200°.*B, (COOH)<sub>2</sub>*: needles. M.p. 105–7°.*Picrate*: needles. M.p. 195–6°.Schulze, Trier, *Z. physiol. Chem.*, 1910, 67, 59; *Ber.*, 1909, 42, 4654.Steenbock, *J. Biol. Chem.*, 1918, 35, 1.**Stachyose** $\text{C}_{24}\text{H}_{42}\text{O}_{21}$ 

MW, 666

Occurs in ash manna, seeds of *Leguminosae*, roots and rhizomes of *Labiates*, e.g., *Stachys tubrifera*. Plates +  $4\text{H}_2\text{O}$  (or  $1\frac{1}{2}\text{H}_2\text{O}$ ). Loses  $\text{H}_2\text{O}$  at  $115^\circ$  in vacuum. M.p. anhyd. 167–70°. Sol.  $\text{H}_2\text{O}$ . Insol. EtOH, Et<sub>2</sub>O. Sweet taste.  $[\alpha]_D^{25} + 133.9^\circ$  in  $\text{H}_2\text{O}$ , +  $148.4^\circ$  (anhyd.) in  $\text{H}_2\text{O}$ . Does not reduce Fehling's. Does not form an osazone. Partly fermentable. Hyd. by dil. acids  $\rightarrow$  glucose (1 mol.) + galactose (2 mols.) + fructose (1 mol.). Heat of comb.  $\text{C}_6 = 3808$  Cal. Forms compounds with BaO and SrO.

*Tetradeca-acetyl*: m.p. 95–6°.  $[\alpha]_D^{25} + 120.2^\circ$  in EtOH,  $[\alpha]_D^{25} + 120.5^\circ$  in AcOH.

*Tetradeca-p-nitrobenzoyl*: m.p. 166°.

*Tetradeca-Me ether*: syrup.  $[\alpha]_D^{24.5} + 133.3^\circ$  in  $C_6H_6$ ,  $+ 139.2^\circ$  in  $CHCl_3$ ,  $+ 129.9^\circ$  in  $Me_2CO$ ,  $+ 135.2^\circ$  in  $EtOH$ ,  $+ 137.1^\circ$  in  $MeOH$ ,  $+ 120.3^\circ$  in  $Et_2O$ .

Tanret, *Compt. rend.*, 1912, **155**, 1526.

Neuberg, Lachmann, *Biochem. Z.*, 1910, **24**, 171.

Onuki, *Chem. Abstracts*, 1932, **26**, 4308; 1933, **27**, 2138.

### Starch (*Amylum*)

$(C_6H_{10}O_5)_n$  MW, (162) $_n$

The main reserve carbohydrate in the vegetable kingdom. Consists of two similarly constituted carbohydrates, amylose and amylopectin, and contains a small percentage of phosphorus. White powder. Insol. cold  $H_2O$ ,  $EtOH$ ,  $Et_2O$ . When heated with water swells and gives an opalescent sol. Hyd. by acids  $\rightarrow$  dextrins  $\rightarrow$  maltose  $\rightarrow$  glucose. Hyd. by diastase  $\rightarrow$  maltose. Gives blue col. with I sol. No generally accepted structure can be given for starch and its derivatives. The following references form a representative selection, which deals with recent work and reviews.

Haworth, *Chemistry and Industry*, 1935, 859.

Kuhn, Ziese, *Ber.*, 1926, **59**, 2314.

Peiser, *Z. physiol. Chem.*, 1926, **161**, 210; 1927, **167**, 88.

Pringsheim, *Ber.*, 1926, **59**, 3008.

Gray, Staud, *Chem. Reviews*, 1927, **4**, 355 (Review).

Hess, Friese, Smith, *Ber.*, 1928, **61**, 1975.

Irvine, *Rec. trav. chim.*, 1929, **48**, 813.

Meyer, Hopff, Mark, *Ber.*, 1929, **62**, 1103.

Baldwin, *J. Am. Chem. Soc.*, 1930, **52**, 2907.

Schoen, *Bull. soc. chim. biol.*, 1930, **12**, 1033 (Review).

v. Náráy-Szabó, *Z. physik. Chem.*, 1930, **151**, 420.

Freudenberg, *J. Soc. Chem. Ind.*, 1931, **50**, 287r (Review).

Haworth, Percival, *J. Chem. Soc.*, 1931, 1342.

Hirst, Plant, Wilkinson, *J. Chem. Soc.*, 1932, 2375.

Samec, *J. Soc. Chem. Ind.*, 1933, **52**, 389r.

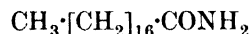
Pringsheim, *Chimie et Industrie (Special Number)*, April 1934, 996 (Review).

Staudinger, Husemann, *Ber.*, 1937, **70**, 1451.

### Stearaldehyde.

See Stearic Aldehyde.

### Stearamide (*Stearic acid amide*)

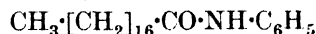


$C_{18}H_{37}ON$  MW, 283

Cryst. from hot  $EtOH$ . M.p. 108.5–109°. B.p. 250°/12 mm. (168–9° in vacuo). Sol.  $Et_2O$ ,  $CHCl_3$ , hot  $EtOH$ . Insol.  $H_2O$ .

Aschan, *Ber.*, 1898, **31**, 2349.

### Stearanilide (*Stearic acid anilide*)



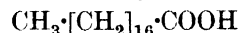
$C_{24}H_{41}ON$  MW, 359

Needles from  $EtOH$ . M.p. 94° (88°). B.p. 153.5°/10 mm. Sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $MeOH$ ,  $Me_2CO$ , hot  $AcOH$ . Spar. sol. pet. ether. Insol.  $H_2O$ .

De' Conno, *Gazz. chim. ital.*, 1917, **47**, i, 98.

Nil, U.S.P., 1,659,150, (*Chem Abstracts*, 1928, **22**, 1367).

### Stearic Acid (*Octadecylic acid*)



$C_{18}H_{36}O_2$  MW, 284

Occurs in moulds, in jalap resin, in fruits of *Citrullus colocynthis*, Schrad., and as esters (glycerides) in fats. Leaflets from  $EtOH$ . F.p. 69.41°. M.p. 71.5–72° (69.6°, 69.5°). B.p. 360° decomp., 291°/100 mm., 238°/17 mm., 232°/15 mm., 158–60°/0.25 mm. Mod. sol. hot  $EtOH$ . Spar. sol.  $C_6H_6$ ,  $CS_2$ .  $D_4^{20}$  0.9408.  $n_D^{20}$  1.4335. Heat of comb. 2711.8 Cal.

*Li salt*: m.p. 220–221.5°.

*Cu salt*: m.p. 115–20°.

*Ag salt*: m.p. 205°.

*Mg salt*: m.p. 132°.

*Pb salt*: m.p. 115.6–115.8° (113–14°).

*Co salt*: m.p. 73–5°.

*Ni salt*: m.p. 80–6°.

*Me ester*:  $C_{19}H_{38}O_2$ . MW, 298. M.p. 38.5–39.5° (39.7–40°). B.p. 442–3°/747 mm., 214–15°/15 mm.

*Et ester*:  $C_{20}H_{40}O_2$ . MW, 312. F.p. 30.92°. (i) M.p. 33.4°. (ii) M.p. 30.9°. B.p. 213–15°/15 mm., 199°/10 mm., 152°/0.18 mm.

*2-Chloroethyl ester*:  $C_{20}H_{39}O_2Cl$ . MW, 346.5. Leaflets from  $EtOH$ . M.p. 49.5°.

*2-Iodoethyl ester*:  $C_{20}H_{39}O_2I$ . MW, 438. Leaflets from  $EtOH$ . M.p. 59.5°.

*Propyl ester*:  $C_{21}H_{42}O_2$ . MW, 326. Prisms from pet. ether. M.p. 28.6°.

*Isobutyl ester*:  $C_{22}H_{44}O_2$ . MW, 340. M.p. 25°. B.p. 199°/5 mm.  $D_4^{20}$  0.8498.  $[\alpha]_D^{20} + 9.39^\circ$  in  $EtOH$ .

*Isoamyl ester*:  $C_{23}H_{46}O_2$ . MW, 354. M.p.  $25.5^\circ$  ( $22^\circ$ ).

*Hexadecyl ester*:  $C_{34}H_{68}O_2$ . MW, 508. M.p.  $55-60^\circ$ .

*Heptadecyl ester*:  $C_{35}H_{70}O_2$ . MW, 522. M.p.  $64.7^\circ$ .

*Ethylene glycol di-ester*:  $C_{38}H_{74}O_4$ . MW, 594. M.p.  $79^\circ$  ( $76^\circ$ ).

*Propylene glycol di-ester*:  $C_{39}H_{76}O_4$ . MW, 608. M.p.  $40^\circ$ .

*Glycerol ester*: see Monostearin, Distearin, Tristearin.

*Phenyl ester*:  $C_{24}H_{40}O_2$ . MW, 360. M.p.  $52^\circ$ . B.p.  $267^\circ/15$  mm.

*o-Nitrophenyl ester*:  $C_{24}H_{39}O_4N$ . MW, 405. M.p.  $60-1^\circ$ .

*p-Tolyl ester*:  $C_{25}H_{42}O_2$ . MW, 374. M.p.  $54^\circ$ . B.p.  $276^\circ/15$  mm.

*Benzyl ester*:  $C_{25}H_{42}O_2$ . MW, 374. M.p.  $28^\circ$ .

*1-Menthyl ester*:  $C_{28}H_{54}O_2$ . MW, 422. Plates from EtOH. M.p.  $39^\circ$ .  $[\alpha]_D^{20} - 36.71^\circ$  in  $CHCl_3$ .

*Chloride*:  $C_{18}H_{35}OCl$ . MW, 302.5. M.p.  $23^\circ$ . B.p.  $202-3^\circ/6$  mm. ( $215^\circ/5$  mm.).

*Amide*: see Stearamide.

*Anhydride*:  $C_{36}H_{70}O_3$ . MW, 550. M.p.  $72^\circ$ .

*Nitrile*: see Stearonitrile.

*Anilide*: see Stearanilide.

*o-Chloroanilide*: m.p.  $67-68.5^\circ$ .

*p-Chloroanilide*: m.p.  $101-2^\circ$ .

*p-Bromoanilide*: m.p.  $114^\circ$ .

*2:4:6-Tribromoanilide*: m.p.  $126^\circ$ .

*p-Nitroanilide*: m.p.  $94.5-95.5^\circ$ .

*o-Toluidide*: m.p.  $97^\circ$ .

*p-Toluidide*: m.p.  $102^\circ$  ( $95.5^\circ$ ). B.p.  $161.5^\circ/10$  mm.

*1-Naphthalide*: m.p.  $110.8^\circ$ . B.p.  $205^\circ/10$  mm.

*2-Naphthalide*: m.p.  $112^\circ$ . B.p.  $239^\circ/10$  mm.

Hell, Sadomski, *Ber.*, 1891, **24**, 2388.

Heintz, *J. prakt. Chem.*, 1855, **66**, 22.

Vesely, *Chimie et industrie*, 1929, **22**, 881 (*Bibl.*).

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 965.

### Stearic Aldehyde (*Stearaldehyde*)



$C_{18}H_{36}O$  MW, 268

M.p.  $55^\circ$  ( $38^\circ$ ). Polymerizes rapidly.

*Oxime*: needles. M.p.  $89^\circ$ .

*Semicarbazone*: needles. M.p.  $108-9^\circ$ .

*Thiosemicarbazone*: needles from EtOH. M.p.  $111^\circ$ .

*p-Nitrophenylhydrazone*: yellow needles from MeOH. M.p.  $101^\circ$ .

Stephen, *J. Chem. Soc.*, 1925, 1876.

Feulgen, Behrens, *Z. physiol. Chem.*, 1928, **177**, 221.

*Note*.—Compounds described in earlier literature are polymers.

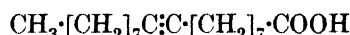
### Stearin.

See Monostearin, Distearin, and Tristearin.

### Stearolactone.

See under 3-Hydroxystearic Acid.

### Stearolic Acid



$C_{18}H_{32}O_2$  MW, 280

Needles from EtOH.Aq. M.p.  $47-8^\circ$ . Sol.  $Et_2O$ , hot EtOH. Insol.  $H_2O$ . Heat of comb.  $C_r$  2624.7 Cal.

*Tetrabromide*: see 8:8:9:9-Tetrabromostearic Acid.

*1-Glycerol ester*:  $C_{21}H_{38}O_4$ . MW, 354. Leaflets from EtOH. M.p.  $40.5^\circ$ .

*1:2-Glycerol di-ester*:  $C_{39}H_{68}O_5$ . MW, 616. M.p.  $40^\circ$ .

*1:3-Glycerol di-ester*: m.p.  $38.5^\circ$ .

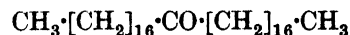
*Glycerol tri-ester*:  $C_{57}H_{98}O_6$ . MW, 878. M.p.  $29^\circ$ .

Hoffmann-La Roche, D.R.P., 243,582, (*Chem. Zentr.*, 1912, I, 695).

Kino, *Chem. Abstracts*, 1930, **24**, 1998; *Chem. Zentr.*, 1936, I, 2531.

See also Posternak, *Compt. rend.*, 1916, **162**, 944.

**Stearone** (*Di-n-heptadecyl ketone*, 18-pentatricontanone)



$C_{35}H_{70}O$  MW, 506

Leaflets from ligroin. M.p.  $88.4^\circ$ . Spar. sol. hot EtOH, hot  $Et_2O$ . Insol.  $H_2O$ .  $D_4^{20}$  0.7932.

*Oxime*: m.p.  $62-3^\circ$ .

Easterfield, Taylor, *J. Chem. Soc.*, 1911, **99**, 2300.

Grün, D.R.P., 296,677, (*Chem. Zentr.*, 1917, I, 611).

**Stearonitrile** (*Stearic acid nitrile*, *heptadecyl cyanide*)



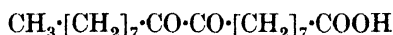
$C_{18}H_{35}N$  MW, 265

M.p. 42.5–43.5°. B.p. 214°/13 mm. (128° in *vacuo*).  $D_4^{20}$  0.8178.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.

van Epps, Reid, *J. Am. Chem. Soc.*, 1916, **38**, 2125.

### Stearoxylic Acid (8 : 9-Diketostearic acid)



$\text{C}_{18}\text{H}_{32}\text{O}_4$  MW, 312

Yellow plates. M.p. 86° (83–4°). Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , hot EtOH. Spar. sol. ligroin.

*Monoxime* : m.p. 76–81°.

*Dioxime* : m.p. 153–4°.

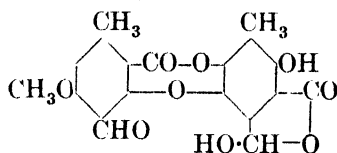
Spieckermann, *Ber.*, 1895, **28**, 276.

Böeseken, *Rec. trav. chim.*, 1911, **30**, 146.

### Stearyl Alcohol.

See Octadecyl Alcohol.

### Stictic Acid (*Scopuloric acid*)



$\text{C}_{19}\text{H}_{14}\text{O}_9$  MW, 386

Occurs in lichen *Ramalina scopulorum*. Needles from  $\text{Me}_2\text{CO}$ . M.p. 270°.  $\text{FeCl}_3 \rightarrow$  purple  $\rightarrow$  brown col.

*Diacetyl deriv.* : m.p. 235–6°.

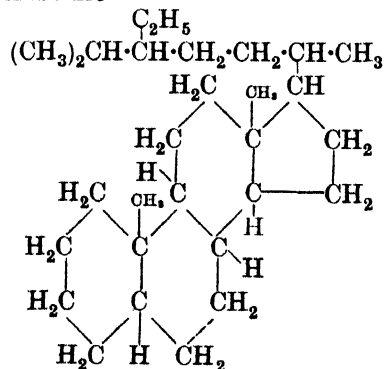
*Tetra-acetyl deriv.* : m.p. 226–7°.

*Di-Me ether* :  $\text{C}_{21}\text{H}_{18}\text{O}_9$ . MW, 414. (i) M.p. 174°. (ii) M.p. 242–3°.

Curd, Robertson, *J. Chem. Soc.*, 1935, 1379.

Asahina *et al.*, *Ber.*, 1933, **66**, 943, 1080; 1936, **69**, 126.

### Stigmastane



$\text{C}_{29}\text{H}_{52}$

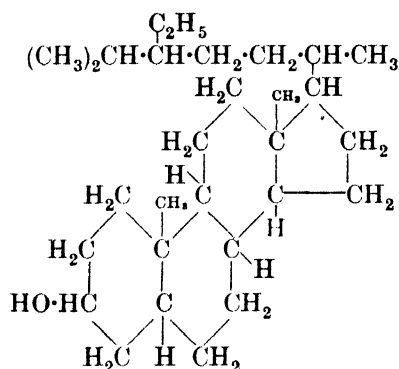
MW, 400

Cryst. from EtOH. M.p. 85.4–85.7°.  $[\alpha]_D^{20} + 25.4^\circ$  in  $\text{CHCl}_3$ . Sol.  $\text{Et}_2\text{O}$ . Spar. sol. EtOH.

Windaus, Brunken, *Z. physiol. Chem.*, 1924, **140**, 47.

See also *Note* under Sitostane.

### Stigmastanol (*Fucostanol*)



$\text{C}_{29}\text{H}_{52}\text{O}$  MW, 416

Plates from EtOH. M.p. 136–7°.  $[\alpha]_D^{20} + 24.8^\circ$  in  $\text{CHCl}_3$ .

*Acetyl* : needles from EtOH. M.p. 130–1°.  $[\alpha]_D^{20} + 15.3^\circ$  in  $\text{CHCl}_3$ .

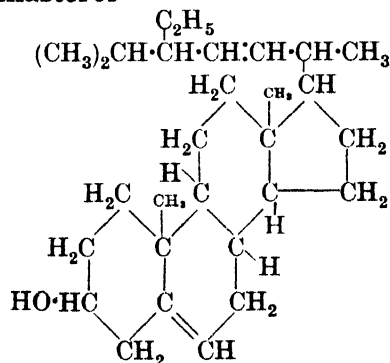
*Benzoyl* : plates from AcOEt. M.p. 135.5–137°.

*3 : 5-Dinitrobenzoyl* : plates from AcOEt. M.p. 214–15°.  $[\alpha]_D^{20} + 13.1^\circ$  in  $\text{C}_6\text{H}_6$ .

Coffey, Heilbron, Spring, *J. Chem. Soc.*, 1936, 738.

See also previous reference and *Note* under Sitostanol.

### Stigmasterol



$\text{C}_{29}\text{H}_{48}\text{O}$  MW, 412

Occurs in soya and calabar beans. Cryst. from EtOH. M.p. 170°.  $[\alpha]_D^{22} - 51.0^\circ$  in  $\text{CHCl}_3$ .

*Acetyl* : plates from EtOH. M.p. 144–144.6°.  $[\alpha]_D^{20} - 55.5^\circ$  in  $\text{CHCl}_3$ . *Tetrabromide* : plates



from EtOH-CHCl<sub>3</sub>. M.p. 211-12° (202-3°).  $[\alpha]_D^{20} - 40^\circ$ .

*Propionyl*: prisms from EtOH. M.p. 122°.

*Palmityl*: m.p. 99°.

*Stearyl*: m.p. 101°.

*Benzoyl*: prisms from CHCl<sub>3</sub>-EtOH. M.p. 160°.

*p-Nitrobenzoyl*: needles from ligroin. M.p. 203°.

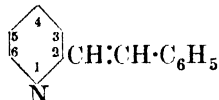
Windaus, Hauth, *Ber.*, 1906, **39**, 4378.

Fernholz, *Ann.*, 1933, **508**, 215.

Guiteras, *Z. physiol. Chem.*, 1933, **214**, 89.

Fernholz, Chakravorty, *Ber.*, 1934, **67**, 2021.

$\alpha$ -Stilbazole (2-Stilbazole, 2-styrylpyridine,  $\beta$ -2-pyridinostyrene)



C<sub>13</sub>H<sub>11</sub>N

MW, 181

Cryst. from EtOH.Aq. M.p. 91°. B.p. 324-5°/750 mm., 194°/14 mm. Sol. Et<sub>2</sub>O, CS<sub>2</sub>. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>, ligroin. Insol. H<sub>2</sub>O. Volatile in steam. KMnO<sub>4</sub>  $\longrightarrow$  benzoic + picolinic acids.

*B,HCl,4H<sub>2</sub>O*: m.p. anhyd. 177°.

*B,HAuCl<sub>4</sub>*: m.p. 185°.

*B,HCl,HgCl<sub>2</sub>*: m.p. 181-3°.

*B,H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 201°.

Wagstaff, *J. Chem. Soc.*, 1934, 277.

Harries, Lenart, *Ann.*, 1915, **410**, 95.

$\gamma$ -Stilbazole (4-Stilbazole, 4-styrylpyridine,  $\beta$ -4-pyridinostyrene).

Prisms, m.p. 131°: leaflets from EtOH, m.p. 127°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O.

*Hydrochloride*: m.p. 204°.

*Hydrobromide*: m.p. 221°.

*Hydriodide*: m.p. 174°.

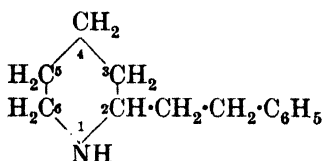
*B,HAuCl<sub>4</sub>*: m.p. 201°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 310°.

*Picrate*: m.p. 113°.

See first reference above.

$\alpha$ -Stilbazoline (2-Phenylethylpiperidine)



C<sub>13</sub>H<sub>19</sub>N

MW, 189

*d*-.

Liq.  $[\alpha]_D^{18} + 12.16^\circ$ .

*Acid d-tartrate*: rhombohedra. M.p. 50°.

*l*-.

Liq.  $[\alpha]_D^{18} - 11.5^\circ$ .

*B,HCl*: needles or prisms from Me<sub>2</sub>CO. M.p. 116-17°.

*Neutral l-tartrate*: plates. M.p. 211-12°.

*Acid d-tartrate*: cryst. + H<sub>2</sub>O. M.p. 78-80°.

*dl*-.

Liq. B.p. 288° (277-8°). D<sub>4</sub> 0.9874. Misc. with EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O with strong alk. reaction.

*B,HCl*: needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 155°.

*B,HBr*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 173°.

*B,HAuCl<sub>4</sub>*: yellow powder. M.p. 133-4°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow powder. M.p. 187-9°.

Ladenburg, *Ber.*, 1904, **37**, 3688.

Baurath, *Ber.*, 1888, **21**, 822.

Bach, *Ber.*, 1901, **34**, 2233.

$\gamma$ -Stilbazoline (4-Phenylethylpiperidine).

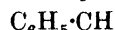
Oil. B.p. 200-10°/80 mm.

*B,HAuCl<sub>4</sub>*: red leaflets. M.p. 204° decomp.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: brown leaflets. M.p. about 210°.

Friedländer, *Ber.*, 1905, **38**, 2837.

Stilbene (sym.-Diphenylethylene, toluylene, dibenzal, dibenzylidene)



Cis (isostilbene)



Trans (stilbene)

C<sub>14</sub>H<sub>12</sub>

MW, 180

*Cis*:

Yellow oil. B.p. 148-9°/17 mm., 145°/13 mm., 136-7°/10 mm.

*Trans*:

Cryst. from EtOH. M.p. 124°. B.p. 305°/720 mm., 166-7°/12 mm. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Shows blue fluor. Volatile in steam. Heat of comb. C<sub>p</sub> 1765.7 Cal.  $n_D^{17} 1.6264$ .

*Picrate*: m.p. 94-5°.

*C<sub>14</sub>H<sub>12</sub>,2SbCl<sub>3</sub>*: m.p. 93°.

*C<sub>14</sub>H<sub>12</sub>,C<sub>6</sub>H<sub>3</sub>(NO<sub>2</sub>)<sub>3</sub>-1:3:5*: m.p. 115-20° (107-10°).

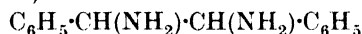
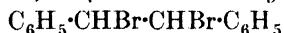
Böeseken, Elsen, *Rec. trav. chim.*, 1928, **47**, 694.

Schenck, Bergmann, *Ann.*, 1928, **463**, 112.

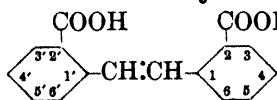
Späth, *Monatsh.*, 1914, **35**, 463.

Corson, E.P., 279,095, (*Chem. Abstracts*, 1928, **22**, 2755).

Ballard, Dehn, *J. Am. Chem. Soc.*, 1932, **54**, 3969.

**Stilbene- $\alpha$ -carboxylic Acid.**See  $\alpha$ -Phenylcinnamic Acid.**Stilbenediamine** ( $\alpha$ :  $\beta$ -Diaminodibenzyl, $\alpha$ :  $\beta$ -diamino-sym.-diphenylethane, diphenylethyl-enediamine) $\text{C}_{14}\text{H}_{16}\text{N}_2$  MW, 212*d*-.  
[ $\alpha$ ]<sub>D</sub> + 134.8°.Feist, Arnstein, *Ber.*, 1895, **28**, 3169.*l*-.  
[ $\alpha$ ]<sub>D</sub> --- 128°.*dl*-.  
Cryst. from ligroin. M.p. 90–2°.*B, HCl, 2H<sub>2</sub>O*: m.p. 251° decomp.*B, H<sub>2</sub>PtCl<sub>6</sub>, 2H<sub>2</sub>O*: m.p. 222–5°.*Picrate*: m.p. 220°.*N*: *N'*-Diacetyl: m.p. above 360°.*N*: *N'*-Dibenzoyl: m.p. 287°.*N*: *N'*-Dibenzylidene: m.p. 152°.*Meso*-.  
Leaflets from Et<sub>2</sub>O. M.p. 121°.*B, 2HCl, 2H<sub>2</sub>O*: m.p. 256° decomp.*B, 2HBr*: m.p. 276–8°.*B, H<sub>2</sub>PtCl<sub>6</sub>*: m.p. 265° decomp.*Picrate*: m.p. 225°.*Dipicrate*: m.p. 239°.*N*: *N'*-Diethyl: m.p. 294°.*N*-Acetyl-*N'*-benzoyl: m.p. 316°.*N*: *N'*-Dibenzoyl: m.p. 350°.*N*: *N'*-Dibenzylidene: m.p. 164°.Biltz, Krebs, *Ann.*, 1912, **391**, 208.Darapsky, Spannagel, *J. prakt. Chem.*, 1915, **92**, 289.**Stilbene dibromide** ( $\alpha$ :  $\beta$ -Dibromo-sym.-diphenylethane,  $\alpha$ :  $\beta$ -dibromodibenzyl) $\text{C}_{14}\text{H}_{12}\text{Br}_2$  MW, 340(i) Needles. M.p. 237°. Sol. hot xylene. Mod. sol. Et<sub>2</sub>O, CS<sub>2</sub>. Spar. sol. EtOH.(ii) Cryst. from hot EtOH. M.p. 111°. Sol. Et<sub>2</sub>O. Spar. sol. EtOH.Liebermann, *Ber.*, 1910, **43**, 1543.Pfeiffer, *Ber.*, 1912, **45**, 1818.Kato, *Chem. Abstracts*, 1932, **26**, 5260.**Stilbene- $\alpha$ :  $\beta$ -dicarboxylic Acid.**

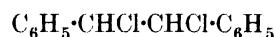
See Diphenylfumaric Acid and Diphenylmaleic Acid.

**Stilbene-2 : 2'-dicarboxylic Acid** $\text{C}_{16}\text{H}_{12}\text{O}_4$ 

Dict. of Org. Comp.—III.

MW, 268

Needles from dil. AcOH. M.p. 263–4° (rapid heat.), 250° (slow heat.).

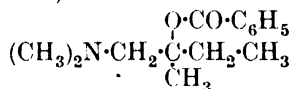
*Di-Et ester*:  $\text{C}_{20}\text{H}_{20}\text{O}_4$ . MW, 324. Needles. M.p. 79–80°. Sol. Et<sub>2</sub>O.Hasselbach, *Ann.*, 1888, **243**, 258.**Stilbene-4 : 4'-dicarboxylic Acid.***Di-Me ester*:  $\text{C}_{18}\text{H}_{16}\text{O}_4$ . MW, 296. Cryst. from EtOH. M.p. 226–7°. Insol. Et<sub>2</sub>O. Sol. in EtOH has bluish-violet fluor.Meyer, Hofmann, *Monatsh.*, 1917, **38**, 358.**Stilbene dichloride** ( $\alpha$ :  $\beta$ -Dichloro-sym.-diphenylethane,  $\alpha$ :  $\beta$ -dichlorodibenzyl) $\text{C}_{14}\text{H}_{12}\text{Cl}_2$  MW, 251

(i) Needles from EtOH. M.p. 191–3°. Sol. hot toluene. Spar. sol. EtOH.

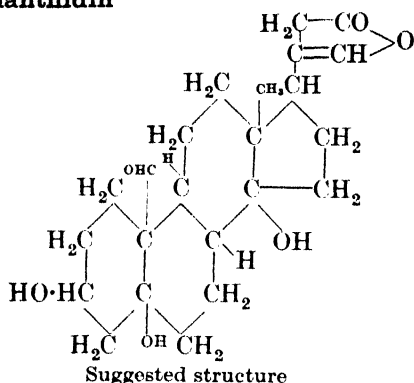
(ii) Leaflets. M.p. 93–4°. Sol. ord. org. solvents. Sublimes.

Pfeiffer, Eistert, *J. prakt. Chem.*, 1930, **124**, 168.Carré, Maucière, *Compt. rend.*, 1931, **192**, 1567.Pfeiffer, *Ber.*, 1912, **45**, 1816.**Stilbene oxide.**

See 1 : 2-Diphenylethylene oxide.

**Stovaine** (1-Dimethylamino-2-methyl-2-butanol benzoate) $\text{C}_{14}\text{H}_{21}\text{O}_2\text{N}$  MW, 235*d*-.  
M.p. 186–7°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 8.5°.*l*-.  
M.p. 186–7°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> — 8.5°.*dl*-.  
M.p. 175°. Local anaesthetic.*B, HCl*: cryst. from EtOH. M.p. 202°.*Picrate*: m.p. 110–12°.Ribas, Rancaño, *Chem. Abstracts*, 1929, **23**, 2164.Fournau, Ribas, *Chem. Abstracts*, 1928, **22**, 761.Fiore, *Chem. Zentr.*, 1916, I, 1076.Riedel, D.R.P., 169,746, (*Chem. Zentr.*, 1906, I, 1584).*Note*.—Both the free base and its hydrochloride are referred to as “Stovaine.”

## Strophanthidin

 $C_{23}H_{32}O_6$ 

MW, 404

Occurs in *Strophanthus kombé*, Oliver. Leaflets +  $2H_2O$  from  $H_2O$ . M.p.  $169-70^\circ$ , anhyd.  $235^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol. pet. ether.  $[\alpha]_D^{25} + 43.1^\circ$  in MeOH.

*Oxime*: m.p.  $270-5^\circ$ .

*Phenylhydrazone*: m.p.  $230-2^\circ$ .

*p*-Bromophenylhydrazone: m.p.  $200^\circ$ .

*Benzoyl*:  $[\alpha]_D^{25} + 47.8^\circ$ .

*p*-Bromobenzoyl: m.p.  $222-4^\circ$ .

Lamb, Smith, *J. Chem. Soc.*, 1936, 444.

Jacobs, Elderfield, *J. Biol. Chem.*, 1935, 108, 497.

Kon, *J. Soc. Chem. Ind.*, 1934, 53, 956.

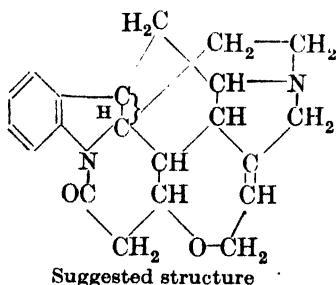
Gamble, Kon, *J. Chem. Soc.*, 1935, 443.

Elderfield, *J. Biol. Chem.*, 1936, 113, 625.

## g-Strophanthin.

See Ouabain.

## Strychnine

 $C_{21}H_{22}O_2N_2$ 

MW, 334

Alkaloid occurring in seeds of *Strychnos nuxvomica*, Linn., *S. ignatii*, Berg. Prisms from EtOH. M.p.  $286-8^\circ$  ( $270-1^\circ$  slow heat.). B.p.  $270^\circ/5$  mm.  $[\alpha]_D^{18} - 139.3^\circ$  in  $CHCl_3$ ,  $-104.5^\circ$  in abs. EtOH. Sol. 6 parts  $CHCl_3$ , 66.6 parts Py, 165 parts  $C_6H_6$ , 6400 parts  $H_2O$  at  $25^\circ$ , and

12 parts boiling 90% EtOH. Prac. insol. abs.  $Et_2O$ . Cold sol. in  $H_2SO_4$  + an oxidising agent e.g.  $K_2C_2O_7$ ,  $KMnO_4$ ,  $PbO_2$ ,  $MnO_2 \rightarrow$  violet-blue col.  $\rightarrow$  purple  $\rightarrow$  red  $\rightarrow$  yellow. Warm in dil.  $HNO_3$  +  $K_2Cr_2O_7 \rightarrow$  intense scarlet col.  $HNO_3$  (D 1.4)  $\rightarrow$  colourless sol.  $\rightarrow$  yellow on heating. Bitter taste. Violent tetanic poison. Employed commercially in vermin killers.

*B, HCl*: needles +  $1\frac{1}{2}H_2O$ .

*B, HBr*: needles +  $H_2O$ . Spar. sol.  $H_2O$ .

*B, HI*: needles +  $H_2O$ . Spar. sol.  $H_2O$ .

*B, HNO\_3*: needles. Sol. 42 parts  $H_2O$  at  $25^\circ$ , 120 parts EtOH at  $25^\circ$ .

*B\_2, H\_2SO\_4*: needles +  $5H_2O$ . M.p. anhyd.  $200^\circ$ . Sol. 48 parts  $H_2O$ , 135 parts EtOH.

*B\_2, H\_2Cr\_2O\_7*: orange-yellow needles. Sol. 1815 parts  $H_2O$  at  $25^\circ$ .

*Monopicrate*: decomp. at  $270^\circ$ .

*Monopicolonate*: decomp. above  $290^\circ$ .

*d-Tartrate*: cryst. +  $7H_2O$ . M.p.  $228^\circ$ .

*l-Tartrate*: cryst. +  $3\frac{1}{2}H_2O$ . M.p.  $242^\circ$ .

*Benzyl chloride*: m.p.  $303.5^\circ$ .

*Benzylidene deriv.*: m.p.  $235-7^\circ$ .

Robinson, *Annual Report of Biochemistry*, 1935, 4, 512 (Bibl.).

Leuchs, Beyer, *Ber.*, 1935, 68, 290.

Clemo, *J. Chem. Soc.*, 1936, 1695.

Kotake, Mori, Mitsuwa, *Sci. Papers Inst. Phys. Chem. Research Tokyo*, 1937, 31, 129.

 $\psi$ -Strychnine $C_{21}H_{23}O_3N_2$ 

MW, 351

A strychnine alkaloid. Cryst. powder. M.p.  $266-8^\circ$  decomp. Mod. sol.  $CHCl_3$ . Spar. sol.  $Me_2CO$ ,  $C_6H_6$ . Insol.  $Et_2O$ .  $[\alpha]_D^{25} - 85.9^\circ$  in  $CHCl_3$ ,  $-43.8^\circ$  in abs. EtOH.  $H_2SO_4$  +  $K_2Cr_2O_7 \rightarrow$  violet col. Not bitter and is less toxic than strychnine.  $Zn + HCl \rightarrow$  strychnine.

*B, HCl*: cryst. +  $2H_2O$  from  $H_2O$ .  $[\alpha]_D^{19} + 3.9^\circ$  in  $H_2O$ ,  $[\alpha]_D^{24} + 8.3^\circ$  in 80% EtOH.

*B, HNO\_3*: spar. sol.  $H_2O$ .  $[\alpha]_D^{25} + 7.6^\circ$  in 80% EtOH.

*B, HClO\_4*: needles from  $H_2O$ . Does not melt below  $300^\circ$ .

*Ferrichloride*: m.p.  $234-5^\circ$ .

*Nitroso deriv.*: cryst. from 80% EtOH. M.p.  $292-4^\circ$ . Sol.  $CHCl_3$ . Spar. sol. MeOH, EtOH,  $Et_2O$ ,  $C_6H_6$ .  $[\alpha]_D^{19} + 223.8^\circ$  in  $CHCl_3$ .

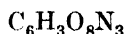
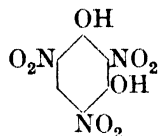
Warnat, *Helv. Chim. Acta*, 1931, 14, 1004.

Blount, Robinson, *J. Chem. Soc.*, 1932, 2305.

## Stygerine.

See 1-Phenylglycerol.

**Styphnic Acid** (2 : 4 : 6-Trinitroresorcinol)



MW, 245

Yellow cryst. from AcOEt. M.p. 179–80° (178°). Explodes on rapid heating. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Forms add. comps. with many hydrocarbons.

*Di-Me ether*:  $C_8H_7O_8N_3$ . MW, 273. Needles from EtOH. M.p. 124–5°.

*Me-Et ether*:  $C_9H_9O_8N_3$ . MW, 287. M.p. 92°.

*Di-Et ether*:  $C_{10}H_{11}O_8N_3$ . MW, 301. Leaflets from EtOH. M.p. 121°.

Brass, Fanta, *Ber.*, 1936, **69**, 6 (*Bibl.*).

Sah, *Chem. Abstracts*, 1932, **26**, 5927.

Vermeulen, *Rec. trav. chim.*, 1919, **38**, 107.

Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2043.

**Stypticin.**

*See under* Cotarnine.

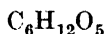
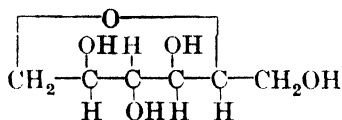
**Styptol.**

*See under* Cotarnine.

**Styracin.**

*See* Cinnamyl cinnamate.

**Styracitol** (1 : 5-Anhydro-d-sorbitol)



MW, 164

Occurs in fruit of *Styrax obassia*. Prisms from 90% EtOH. M.p. 157° (155°). Sol. H<sub>2</sub>O. Spar. sol. cold EtOH. Prac. insol. Et<sub>2</sub>O, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Bitter-sweet taste.  $[\alpha]_D^{20} - 71.72^\circ$  in H<sub>2</sub>O ( $[\alpha]_D^{17} - 49.4^\circ$  in H<sub>2</sub>O). Does not reduce Fehling's.

*Tetra-acetyl*: prisms from H<sub>2</sub>O, m.p. 66–7°; needles from EtOH, m.p. 58°. B.p. 200°/7 mm.  $[\alpha]_D^{22} - 20.86^\circ$  in EtOH.

*Tetrazenzoyl*: m.p. 142°.

*Tetra-Me ether*: b.p. 149–51°/24 mm., 143–4°/16 mm.  $D_4^{18} 1.1092$ .  $n_D^{14} 1.4516$ .  $[\alpha]_D^{14} - 35.63^\circ$ .

*Diacetone deriv.*: m.p. 96–7°.  $[\alpha]_D^{17} - 115.24^\circ$  in EtOH.

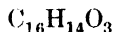
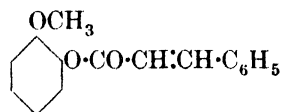
*Dibenzylidene deriv.*: (i) m.p. 163–5°.  $[\alpha]_D^{25} - 148.73^\circ$  in CHCl<sub>3</sub>; (ii) m.p. 192–3°.  $[\alpha]_D^{25} - 80.47^\circ$  in CHCl<sub>3</sub>.

Asahina, *Ber.*, 1912, **45**, 2363.

Asahina, Takimoto, *Ber.*, 1931, **64**, 1803.

Zervas, *Ber.*, 1930, **63**, 1689.

**Styracol** (*Guaiacol cinnamate*)

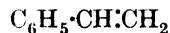


MW, 254

Needles from EtOH. M.p. 130°.

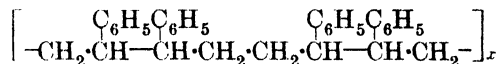
Knoll, D.R.P., 62, 176.

**Styrene** (*Styrol*, *styrolene*, *cinnamol*, *cinnamene*, *vinylbenzene*, *phenylethylene*)



MW, 104

Occurs in storax. F.p. – 33°. M.p. – 33°. B.p. 145–145.8°, 52–3°/28 mm., 48°/20 mm., 40°/14 mm., 33°/10 mm. Sol. EtOH, Et<sub>2</sub>O, MeOH, Me<sub>2</sub>CO, CS<sub>2</sub>. Spar. sol. H<sub>2</sub>O.  $D_4^{20} 0.9090$ .  $n_D^{20} 1.5462$ . Polymerises slowly, rapidly in presence of metallic sodium, to



*Metastyrene*:  $(C_8H_8)_x$ . MW, (104)<sub>x</sub>. Amorphous mass. Spar. sol. hot Et<sub>2</sub>O. Insol. H<sub>2</sub>O, EtOH. Irradiation in C<sub>6</sub>H<sub>6</sub> or dist. → styrene.

*Polystyrene*: a rubber-like substance.

*Tetrazyrene*:  $C_{32}H_{32}$ . MW, 416. B.p. 210–17°/0.05 mm.

Naugatuck, D.R.P., 600,268, (*Chem. Abstracts*, 1934, **28**, 6448).

Sontag, *Ann. chim.*, 1934, I, 359.

Zal'kind, Bulavskii, *Chem. Abstracts*, 1936, **30**, 1368.

Gibbons, Smith, U.S.P., 1,938,827, (*Chem. Abstracts*, 1934, **28**, 1367).

Houtz, Adkins, *J. Am. Chem. Soc.*, 1933, **55**, 1609.

Gautier, Gautier, *Bull. soc. chim.*, 1933, **53**, 323.

Midgley, Henne, Leicester, *J. Am. Chem. Soc.*, 1936, **58**, 1961.

Graves, U.S.P., 2,036,410, (*Chem. Abstracts*, 1936, **30**, 3446).

Waterman, Kok, *Rec. trav. chim.*, 1934, **53**, 1133 (*Bibl.*).

I.G., U.S.P., 2,005,295, (*Chem. Abstracts*, 1935, **29**, 5203).

Staudinger, D.R.P., 610,478, (*Chem. Abstracts*, 1935, **29**, 3752).

Hibbert, Burt, *J. Am. Chem. Soc.*, 1925, **47**, 2240.

**Styrene bromohydrin** (*Phenylethylene bromohydrin*,  $\beta$ -bromo- $\alpha$ -hydroxyethylbenzene, bromomethylphenylcarbinol)

$C_6H_5 \cdot CH(OH) \cdot CH_2Br$   
 $C_8H_9OBr$  MW, 201  
 B.p.  $109-10^\circ/2$  mm.  $n_D^{17}$  1.5800.  $D_4^{20}$  1.4994.  
 Read, Reid, *J. Chem. Soc.*, 1928, 1488.

**Styrene chlorohydrin** (*Phenylethylene chlorohydrin*,  $\beta$ -chloro- $\alpha$ -hydroxyethylbenzene, chloromethylphenylcarbinol)

$C_6H_5 \cdot CH(OH) \cdot CH_2Cl$   
 $C_8H_9OCl$  MW, 156.5  
 B.p.  $128^\circ/17$  mm.  $D_4^0$  1.225.  $n_D^{17}$  1.55405.  
 Detoeuf, *Bull. soc. chim.*, 1922, 31, 176.

**Styrene dibromide** ( $\alpha$  :  $\beta$ -Dibromoethylbenzene, phenylethylene dibromide)

$C_6H_5 \cdot CHBr \cdot CH_2Br$   
 $C_8H_8Br_2$  MW, 264  
 Leaflets or needles from EtOH.Aq. M.p.  $74-74.5^\circ$  ( $72-3^\circ$ ). B.p.  $139-41^\circ/15$  mm. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , AcOH, ligroin.

Evans, Morgan, *J. Am. Chem. Soc.*, 1913, 35, 56.

Glaser, *Ann.*, 1870, 154, 154.

Zincke, *Ann.*, 1883, 216, 288.

**Styrene dichloride** ( $\alpha$  :  $\beta$ -Dichloroethylbenzene, phenylethylene dichloride)

$C_6H_5 \cdot CHCl \cdot CH_2Cl$   
 $C_8H_8Cl_2$  MW, 175  
 B.p.  $233-4^\circ/759$  mm.,  $114.5-115.5^\circ/15$  mm.  $D_4^{15}$  1.240.  $n_D^{15}$  1.5544.

Biltz, *Ann.*, 1897, 296, 275.

**Styrene Glycol** (*Phenylethylene glycol*,  $\alpha$  :  $\beta$ -dihydroxyethylbenzene)

$C_6H_5 \cdot CH(OH) \cdot CH_2OH$   
 $C_8H_{10}O_2$  MW, 138  
 Needles from ligroin. M.p.  $67-8^\circ$ . B.p.  $272-4^\circ/755$  mm. Very sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ . Spar. sol. ligroin.

*Me ether* :  $C_9H_{12}O_2$ . MW, 152. B.p.  $237-8^\circ$ ,  $132^\circ/18$  mm.  $D_4^0$  1.080.

*Et ether* :  $C_{10}H_{14}O_2$ . MW, 166. B.p.  $242-3^\circ$ .  $D_4^0$  1.054.

*Di-Et ether* :  $C_{12}H_{18}O_2$ . MW, 194. B.p.  $105-6^\circ/10$  mm. Dil.  $H_2SO_4 \rightarrow$  phenylacetaldehyde.

*Di-formyl* : b.p.  $164-5^\circ/25$  mm.  $D_4^0$  1.2091.

*Diacetyl* : b.p.  $274^\circ/755$  mm.,  $183-5^\circ/25$  mm.

*Dibenzoyl* : needles from EtOH or toluene. M.p.  $96-7^\circ$ . Sublimes.

Zincke, *Ann.*, 1883, 216, 293.

Tiffeneau, *Compt. rend.*, 1907, 145, 812.

Späth, *Monatsh.*, 1914, 35, 332.

Balla, *Compt. rend.*, 1934, 198, 948.

**Styrene oxide** (*Phenylethylene oxide*)

$C_6H_5 \cdot CH-CH_2$   
 $\diagup \quad \diagdown$   
 $O$   
 $C_8H_8O$  MW, 120

Liq. with aromatic odour. B.p.  $191-2^\circ$ ,  $84-5^\circ/15$  mm.,  $77.5-78.5^\circ/12$  mm.  $D_4^{15}$  1.0523.

Fourneau, Tiffeneau, *Compt. rend.*, 1905, 140, 1595; 1908, 146, 697.

Hibbert, Burt, *Organic Syntheses*, Collective Vol. I, 481.

**Styrol.**

See Styrene.

**Styrolene.**

See Styrene.

**Styrone.**

Cinnamaldehyde, *q.v.*

**Styrylacetic Acid** (3-Phenylvinylacetic acid, 2-benzylidenepropionic acid)

$C_6H_5 \cdot CH : CH \cdot CH_2 \cdot COOH$   
 $C_{10}H_{10}O_2$  MW, 162

I.

Needles from  $H_2O$ . M.p.  $87^\circ$ . B.p.  $302^\circ$ . Sol. EtOH,  $Et_2O$ , hot  $CS_2$ .

*K salt* : m.p.  $245^\circ$  decomp.

*Me ester* :  $C_{11}H_{12}O_2$ . MW, 176. B.p.  $185^\circ$  / about 20 mm.

*Et ester* :  $C_{12}H_{14}O_2$ . MW, 190. B.p.  $281-2^\circ$  /  $763$  mm.,  $184^\circ/44$  mm.,  $164-5^\circ/25$  mm.  $D_4^{20}$  1.0340.  $n_D^{20}$  1.53271.

*Chloride* :  $C_{10}H_9OCl$ . MW, 180.5. M.p.  $41-2^\circ$ . B.p.  $106-8^\circ/0.4$  mm.

*Amide* :  $C_{10}H_{11}ON$ . MW, 161. Needles from  $C_6H_6$ . M.p.  $130^\circ$ . Sol. EtOH,  $CHCl_3$ , hot  $H_2O$ . Spar. sol.  $Et_2O$ .

*Anhydride* :  $C_{20}H_{18}O_3$ . MW, 306. M.p.  $120-1^\circ$ .

*Nitrile* : cinnamyl cyanide.  $C_{10}H_9N$ . MW, 143. Leaflets from pet. ether. M.p.  $61-2^\circ$ .

II. *Allo.*

*Amide* : needles from  $C_6H_6$ -pet. ether. M.p.  $85-6^\circ$ .

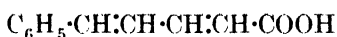
Stoermer, Stockmann, *Ber.*, 1914, 47, 1794.

Borsche, Niemann, Hartman, *Ber.*, 1936, 69, 1996.

Linstead, Williams, *J. Chem. Soc.*, 1926, 2741.

Fichter, Pfister, *Ber.*, 1904, 37, 2001.

**2-Styrylacrylic Acid** (*Cinnamylideneacetic acid, 4-phenyl-1:3-butadiene-1-carboxylic acid, 4-phenylvinylacrylic acid*)



$\text{C}_{11}\text{H}_{10}\text{O}_2$

MW, 174

$\alpha$ -.

Plates from EtOH, prisms from  $\text{C}_6\text{H}_6$ . M.p. 165–6°. Sol. EtOH. Spar. sol. pet. ether. Heat of comb.  $C_p$  1312.7 Cal,  $C_r$  1311.8 Cal.

$A, \text{C}_6\text{H}_5\cdot\text{NH}_2$ : m.p. 142–3°.

$A, \text{C}_2\text{H}_5\cdot\text{NH}_2$ : m.p. 117–18°.

$A, (\text{C}_2\text{H}_5)_2\text{NH}$ : m.p. about 80°.

*Me ester*:  $\text{C}_{12}\text{H}_{12}\text{O}_2$ . MW, 188. M.p. 71°. B.p. 185°/20 mm.

*Et ester*:  $\text{C}_{13}\text{H}_{14}\text{O}_2$ . MW, 202. Oil.  $D_4^{20}$  0.9985.

*Isopropyl ester*:  $\text{C}_{14}\text{H}_{16}\text{O}_2$ . MW, 216. Yellow oil. B.p. 169°/9 mm.  $D_4^{20}$  1.0256.  $n_D^{19.7}$  1.6066.

*Amide*:  $\text{C}_{11}\text{H}_{11}\text{ON}$ . MW, 173. Leaflets from  $\text{C}_6\text{H}_6$ . M.p. 185°. *Acetyl*: m.p. 177–8°.

*Methylamide*:  $\text{C}_{12}\text{H}_{13}\text{ON}$ . MW, 187. Needles from  $\text{C}_6\text{H}_6$ –EtOH. M.p. 157°.

*Ethylamide*:  $\text{C}_{13}\text{H}_{15}\text{ON}$ . MW, 201. Needles from  $\text{C}_6\text{H}_6$ . M.p. 143–4°.

*Diethylamide*:  $\text{C}_{15}\text{H}_{19}\text{ON}$ . MW, 229. M.p. 106°.

*Anhydride*:  $\text{C}_{22}\text{H}_{18}\text{O}_3$ . MW, 330. Needles from AcOH. M.p. 152°.

*Nitrile*:  $\text{C}_{11}\text{H}_9\text{N}$ . MW, 155. Cryst. from ligroin. M.p. 42°. B.p. 285°, 159°/285 mm.

$\beta$ -. *Allo*-.

Needles from  $\text{C}_6\text{H}_6$ . M.p. 138°. Sol. Et<sub>2</sub>O, CS<sub>2</sub>, CHCl<sub>3</sub>, hot  $\text{C}_6\text{H}_6$ . Heat of comb.  $C_p$  1321.6 Cal,  $C_r$  1320.7 Cal.

*Me ester*: f.p. – 15°. Decomp. on dist.

Riiber, *Ber.*, 1911, **44**, 2390; *Ber.*, 1904, **37**, 2274.

Reynolds, *Am. Chem. J.*, 1911, **46**, 200.

Stobbe, *Ber.*, 1912, **45**, 3408.

**Styryl Alcohol.**

See Cinnamyl Alcohol.

**Styryl bromide.**

See  $\beta$ -Bromostyrene.

**1-Styryl-1-butylene.**

See 1-Phenyl-1:3-hexadiene.

**Styryl chloride.**

See  $\beta$ -Chlorostyrene.

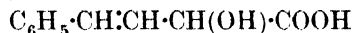
**Styryl cyanide.**

See under Cinnamic Acid.

**2-Styryl-1-cyanoacrylic Acid.**

See Cinnamylidenecyanoacetic Acid.

**Styrylglycollic Acid** (*1-Hydroxy-3-phenylvinylacetic acid, 1-hydroxy-2-benzylidenepropionic acid, benzylidene-lactic acid*)



$\text{C}_{10}\text{H}_{10}\text{O}_3$

MW, 178

Needles from H<sub>2</sub>O. M.p. 137°. Very sol. boiling H<sub>2</sub>O. Spar. insol. H<sub>2</sub>O, Et<sub>2</sub>O. Insol. CS<sub>2</sub>,  $\text{C}_6\text{H}_6$ , ligroin.

*Amide*:  $\text{C}_{10}\text{H}_{11}\text{O}_2\text{N}$ . MW, 177. Plates from H<sub>2</sub>O. M.p. 141.5°. Very sol. EtOH. Sol. hot CHCl<sub>3</sub>,  $\text{C}_6\text{H}_6$ . Spar. sol. Et<sub>2</sub>O, CS<sub>2</sub>.

*Nitrile*: cinnamaldehyde cyanhydrin.  $\text{C}_{10}\text{H}_9\text{ON}$ . MW, 159. Plates from CS<sub>2</sub>. M.p. 74°. *Benzoyl*: cryst. from H<sub>2</sub>O. M.p. 72–3°.

*Acetyl*: needles from H<sub>2</sub>O. M.p. 79° (+ 1H<sub>2</sub>O), 90–1° (anhyd.). Very sol. most org. solvents.

Fittig, Ginsberg, Petkow, *Ann.*, 1898, **299**, 20.

Thiele, Sulzberger, *Ann.*, 1901, **319**, 208.

Bougault, *J. pharm. chim.*, 1913, **8**, 404.

**Styrylphenol.**

See Hydroxystilbene.

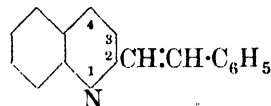
**2-Styrylpropionic Acid.**

See 3-Benzylidenebutrylic Acid.

**1-Styrylpyridine.**

See Stilbazole.

**2-Styrylquinoline** (*2-Benzylidenequinaldine, sym.-phenyl-2-quinolylethylene,  $\alpha$ -irazole*)



$\text{C}_{17}\text{H}_{13}\text{N}$

MW, 231

Needles from Et<sub>2</sub>O–EtOH. M.p. 99–100°. Sol. CHCl<sub>3</sub>, CS<sub>2</sub>, hot EtOH. Insol. H<sub>2</sub>O.  $\text{CrO}_3 \rightarrow$  benzoic + quinaldinic acids.

Ismailsky, *J. prakt. Chem.*, 1912, **85**, 91.

Noelting, Witte, *Ber.*, 1906, **39**, 2750.

**4-Styrylquinoline** ( *$\omega$ -Benzylidenelepidine, sym.-phenyl-4-quinolylethylene*).

Cryst. from EtOH. M.p. 92°. Sol. Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.  $\text{CrO}_3 \rightarrow$  cinchoninic acid.

Doebner, Miller, *Ber.*, 1885, **18**, 1646.

**Suberamic Acid.**

See under Suberic Acid.

**Suberane.**

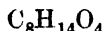
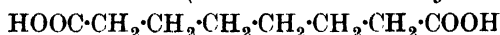
See Cycloheptane.

**Suberanilic Acid.**

See under Suberic Acid.

**Suberene.**

See Cycloheptene.

**Suberic Acid** (*Hexane-1:6-dicarboxylic acid*)

MW, 174

Needles from  $\text{H}_2\text{O}$ . M.p.  $144^\circ$  ( $139\text{--}40^\circ$ ). B.p.  $279^\circ/100$  mm.,  $258.5^\circ/50$  mm.,  $230^\circ/15$  mm.,  $219.5^\circ/10$  mm. Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{CHCl}_3$ . Heat of comb.  $\text{C}_p$  982.8 Cal.,  $\text{C}_r$  988.6 Cal.  $k$  (first) =  $2.58 \times 10^{-5}$  at  $25^\circ$ ; (second) =  $2.5 \times 10^{-6}$  at  $100^\circ$ .

*Di-brucine salt*: m.p.  $102^\circ$ .

*Me ester*:  $\text{C}_9\text{H}_{16}\text{O}_4$ . MW, 188. F.p. about  $10^\circ$ . B.p.  $146\text{--}50^\circ/1$  mm.  $D_4^{20}$  1.047.

*Di-Me ester*:  $\text{C}_{10}\text{H}_{18}\text{O}_4$ . MW, 202. F.p.  $-3.1^\circ$ . B.p.  $268^\circ$ ,  $174\text{--}5^\circ/13\text{--}20$  mm.,  $130\text{--}1^\circ/9$  mm.  $D_4^{20}$  1.0217.  $n_D^{20}$  1.43408.

*Et ester*:  $\text{C}_{10}\text{H}_{18}\text{O}_4$ . MW, 202. M.p.  $21\text{--}2^\circ$  ( $n_5^\circ$ ). B.p.  $186\text{--}188.5^\circ/16$  mm.  $D_4^{25}$  1.037.  $n_D^{25}$  1.4412. *Chloride*:  $\text{C}_{10}\text{H}_{17}\text{O}_3\text{Cl}$ . MW, 220.5. B.p.  $132^\circ/15$  mm. *p-Toluidide*: m.p.  $74^\circ$ .

*Di-Et ester*:  $\text{C}_{12}\text{H}_{22}\text{O}_4$ . MW, 230. F.p.  $5.9^\circ$ . B.p.  $282^\circ/763$  mm.,  $251\text{--}3^\circ/320$  mm.  $D_4^{20}$  0.9822.  $n_D^{20}$  1.43278.

*Phenacyl ester*:  $\text{C}_{16}\text{H}_{20}\text{O}_5$ . MW, 292. M.p.  $102.4^\circ$ .

*p-Bromophenacyl ester*:  $\text{C}_{16}\text{H}_{19}\text{O}_5\text{Br}$ . MW, 371. M.p.  $144.2^\circ$ .

*Dichloride*:  $\text{C}_8\text{H}_{12}\text{O}_2\text{Cl}_2$ . MW, 211. B.p.  $162\text{--}3^\circ/15$  mm.,  $149\text{--}50^\circ/12$  mm.  $D_4^{20.8}$  1.1718.  $n_D^{20.6}$  1.46847.

*Amide*: suberamic acid.  $\text{C}_8\text{H}_{15}\text{O}_3\text{N}$ . MW, 173. Cryst. from  $\text{H}_2\text{O}$ . M.p.  $125\text{--}7^\circ$ .

*Diamide*:  $\text{C}_8\text{H}_{16}\text{O}_2\text{N}_2$ . MW, 172. Cryst. from  $\text{H}_2\text{O}$ . M.p.  $216\text{--}17^\circ$ .

*Anhydride*: suberic anhydride.  $\text{C}_8\text{H}_{12}\text{O}_3$ . MW, 156. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $65\text{--}6^\circ$  ( $63^\circ$ ).

*Dinitrile*: suberonitrile.  $\text{C}_8\text{H}_{12}\text{M}_2$ . MW, 136. M.p.  $-3.5^\circ$ . B.p.  $185^\circ/15$  mm.,  $176\text{--}8^\circ/11$  mm.

*Anilide*: suberanilic acid. M.p.  $128^\circ$ .

*Dianilide*: m.p.  $187^\circ$  ( $183^\circ$ ).

*Di-p-toluidide*: m.p.  $218^\circ$ .

*Dihydrazide*: m.p.  $185\text{--}6^\circ$ .

Green, Hilditch, *J. Chem. Soc.*, 1937, 766.  
Rozanov, Belikov, *Chem. Abstracts*, 1930, 24, 3765.

Verkade, *Rec. trav. chim.*, 1927, 46, 137.

Arppe, *Ann.*, 1862, 124, 89.

Dale, Schorlemmer, *Ann.*, 1879, 199, 145.

Blaise, Koehler, *Bull. soc. chim.*, 1909, 5, 690.

**Suberol.**

See Cycloheptanol.

**Suberone.**

See Cycloheptanone.

**Suberyl Alcohol.**

See Cycloheptanol.

**Suberylamine.**

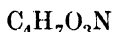
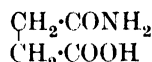
See Cycloheptylamine.

**Suberylene.**

See Cycloheptene.

**Succinaldehydic Acid.**

2-Aldehydopropionic Acid, *q.v.*

**Succinamic Acid** (*Succinic acid monoamide*)

MW, 117

Needles from  $\text{Me}_2\text{CO}$ . M.p.  $157^\circ$  ( $154^\circ$ ). Mod. sol.  $\text{H}_2\text{O}$ , hot  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{EtOH}$ .  $\text{C}_6\text{H}_6$ , ligroin. Heat at  $200^\circ \rightarrow$  succinimide.

*A.NH<sub>2</sub>.NH<sub>2</sub>*: m.p.  $113^\circ$ .

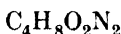
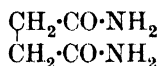
*Me ester*:  $\text{C}_5\text{H}_9\text{O}_3\text{N}$ . MW, 131. Plates. M.p.  $89\text{--}91^\circ$ . Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{Et}_2\text{O}$ , pet. ether.

*Et ester*:  $\text{C}_6\text{H}_{11}\text{O}_3\text{N}$ . MW, 145. M.p.  $74\text{--}5^\circ$ . B.p.  $230\text{--}40^\circ$ .

*Amide*: see Succinamide.

Rubtzov, *Chem. Abstracts*, 1924, 18, 1472.

Jeffery, Vogel, *J. Chem. Soc.*, 1934, 1103.

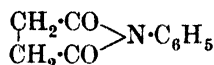
**Succinamide** (*Succinic acid diamide*)

MW, 116

Needles from  $\text{H}_2\text{O}$ . M.p.  $260^\circ$  decomp. ( $242.3^\circ$ ). Sol. hot  $\text{H}_2\text{O}$ . Insol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Heat of comb.  $\text{C}_p$  509.7 Cal. Heat  $\rightarrow$  succinimide.

*N-d-Glucoside*:  $\text{C}_{10}\text{H}_{18}\text{O}_7\text{N}_2$ . MW, 278. Prisms +  $2\text{H}_2\text{O}$  from  $\text{EtOH.Aq}$ . M.p.  $88\text{--}90^\circ$  ( $192^\circ$  decomp., anhyd.).  $[\alpha]_D^{20} - 17.40^\circ$  in  $\text{H}_2\text{O}$ .

Morrell, *J. Chem. Soc.*, 1914, 105, 2705.

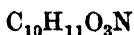
**Succinanil (N-Phenylsuccinimide)**

MW, 175

Needles from  $\text{H}_2\text{O}$ . M.p.  $156^\circ$ . Distills at about  $400^\circ$  without decomp. Sol.  $\text{Et}_2\text{O}$ , hot  $\text{EtOH}$ . Insol.  $\text{H}_2\text{O}$ .

Warren, Briggs, *Ber.*, 1931, 64, 29.

Ruggli, *Ann.*, 1916, 412, 4.

**Succinanilic Acid** (*Succinic acid mono-anilide*)

MW, 193

Needles from hot  $\text{H}_2\text{O}$ . M.p. 144.5–145.5° (148.5°). Sol. EtOH,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Heat of comb.  $C_p$  1166.5 Cal.  $k = 2.03 \times 10^{-5}$  at 25°. Heat  $\rightarrow$  succinanil.

$A, CH_3 \cdot NH_2$ : m.p. 115–20°.

*Me ester*:  $C_{11}H_{13}O_3N$ . MW, 207. Needles from EtOH. M.p. 97–8°.

*Et ester*:  $C_{12}H_{15}O_3N$ . MW, 221. Cryst. from  $\text{Et}_2\text{O}$ -pet. ether. M.p. 56.5–57.5°.

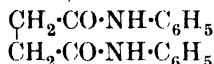
*Amide*:  $C_{10}H_{12}O_2N_2$ . MW, 192. Needles from  $\text{H}_2\text{O}$ . M.p. 181°. Heat of comb.  $C_p$  1244.6 Cal.

*Anilide*: see Succinanilide.

Menschutkin, *Ann.*, 1872, **162**, 176.

See also Warren, Briggs, *Ber.*, 1931, **64**, 29.

**Succinanilide** (*Succinic acid dianilide*,  $N:N'$ -diphenylsuccinamide)



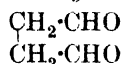
$C_{16}H_{16}O_2N_2$  MW, 268

Needles from EtOH. M.p. 230°. Sol.  $\text{Et}_2\text{O}$ , hot EtOH. Insol.  $\text{H}_2\text{O}$ . Heat. of comb  $C_p$  1971.3 Cal. Dist.  $\rightarrow$  succinanil + aniline.

Morrell, *J. Chem. Soc.*, 1914, **105**, 1736, 2702.

Mistry, Guha, *J. Indian Chem. Soc.*, 1930, **I**, 797.

**Succindialdehyde** (*Succinic dialdehyde*, *succinic aldehyde*, *dialdehydoethane*)



$C_4H_6O_2$  MW, 86

Only polymers produced in preparation. Heated in vacuo  $\rightarrow$  monomer. B.p. 169–70° decomp., 56.5°/9 mm.  $D_4^{18}$  1.069.  $n_D^{18}$  1.42617.

*Monoxime*: *Me ether*, b.p. 67°/10 mm.

*Dioxime*: m.p. 173°.

*Di-[di-Me]-acetal*: b.p. 201–2°/772 mm., 99°/22 mm., 89°/13 mm.  $n_D^{20}$  1.41555.

*Di-[di-Et]-acetal*: b.p. 210–15° decomp., 137°/35 mm., 116°/20 mm.

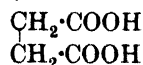
*Polymer*: m.p. 65°. B.p. 169°.

Harries, *Ann.*, 1913, **395**, 260.

Harries, Hohenemser, *Ber.*, 1908, **41**, 255 (Footnote 4).

Schöpf, Lehmann, *Ann.*, 1935, **518**, 1.

**Succinic Acid** (*Ethane-1:2-dicarboxylic acid*)



$C_4H_6O_4$  MW, 118

Occurs in fossils, algæ, lichens, fungi, etc. Prisms from  $\text{H}_2\text{O}$ . M.p. 185° (184.5–185°). B.p. 235°  $\rightarrow$  anhydride. Sol. EtOH, MeOH,  $\text{Me}_2\text{CO}$ , hot  $\text{H}_2\text{O}$ , hot  $\text{H} \cdot \text{COOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ .  $k$  (first) =  $6.37 \times 10^{-3}$  ( $6.6 \times 10^{-5}$ ) at 25°; (second) =  $2.54 \times 10^{-6}$  ( $2.7 \times 10^{-6}$ ) at 25°.

$A, 2NH_2OH$ : m.p. 121° decomp.

*Brucine salt*: m.p. 216–18°.  $[\alpha]_D^{20} - 25.4^\circ$  in  $\text{H}_2\text{O}$ .

*Quinine salt*: m.p. 198–200°.  $[\alpha]_D^{20} - 165.5^\circ$  in  $\text{H}_2\text{O}$ .

*Strychnine salt*: m.p. 210°.  $[\alpha]_D^{20} - 25.6^\circ$  in  $\text{H}_2\text{O}$ .

*Me ester*:  $C_5H_8O_4$ . MW, 132. M.p. 58°. B.p. 151°/20 mm., 121–3°/4 mm.

*Di-Me ester*: see Dimethyl succinate.

*Et ester*:  $C_6H_{10}O_4$ . MW, 146. M.p. 8°. B.p. 172°/42 mm., 146–9°/17 mm., 119°/3 mm.  $D^{20}$  1.1466.  $n_D^{20}$  1.4327. *Chloride*:  $C_6H_8O_3Cl$ . MW, 163.5. B.p. 144°/90 mm., 92°/20 mm.

*Me-Et ester*:  $C_7H_{12}O_4$ . MW, 160. F.p. below –20°. B.p. 208–2°.

*Di-Et ester*: see Diethyl succinate.

*Di-2-hydroxyethyl ester*:  $C_8H_{14}O_6$ . MW, 206. B.p. 176–80°/0.01 mm. *p-Nitrobenzoyl deriv.*: m.p. 90–1°. *Diphenylurethane*: m.p. 113°.

*Di-2-chloroethyl ester*:  $C_8H_{12}O_4Cl_2$ . MW, 243. B.p. 204–5°/30 mm.

*Propyl ester*:  $C_7H_{12}O_4$ . MW, 160. M.p. 15°. B.p. 126°/3 mm.  $D^{20}$  1.1071.  $n_D^{20}$  1.4343.

*Dipropyl ester*:  $C_{10}H_{18}O_4$ . MW, 202. M.p. –10.4°. B.p. 250.8° (246–7°/762.2 mm.).  $D^{20}$  1.0011.  $n_D^{20}$  1.4252.

*Di-isopropyl ester*: b.p. 228°/761 mm.

*Butyl ester*:  $C_8H_{14}O_4$ . MW, 174. M.p. 8.6°. B.p. 136.5°/3 mm.  $D^{20}$  1.0732.  $n_D^{20}$  1.4360.

*Dibutyl ester*:  $C_{12}H_{22}O_4$ . MW, 230. M.p. –29.25° (–37.5°). B.p. 274.5°, 145°/4 mm.  $D^{20}$  0.9652.  $n_D^{20}$  1.4369.

*Di-sec. n-butyl ester*: b.p. 255.5–256.5°/750 mm.  $D^{20}$  0.9735.

*Di-isobutyl ester*: b.p. 264.8–265.8°.

*n-Amyl ester*:  $C_9H_{16}O_4$ . MW, 188. M.p. 17.2°. B.p. 147°/3 mm.  $D^{20}$  1.0460.  $n_D^{20}$  1.4378.

*Di-n-amyl ester*:  $C_{14}H_{26}O_4$ . MW, 258. M.p. –9°. B.p. 171.5°/16 mm.

*Di-active-amyl ester*: b.p. 175°/20 mm.

*Di-isoamyl ester*: b.p. 298–9°/765.4 mm., 289.9°/728 mm.

*Di-n-heptyl ester*:  $C_{18}H_{34}O_4$ . MW, 314. B.p. 350–1°.

*Di-hexadecyl ester*:  $C_{36}H_{70}O_4$ . MW, 566. M.p. 58°.

*Diallyl ester*:  $C_{10}H_{14}O_4$ . MW, 198. B.p. 249–50°/757.3 mm.



*Cyclohexyl ester*:  $C_{10}H_{16}O_4$ . MW, 200. M.p. 44°.

*d-Menthyl ester*:  $C_{14}H_{24}O_4$ . MW, 256.  $[\alpha]_D + 55.68^\circ$  in  $C_6H_6$ . *Cinchonidine salt*: m.p. 141–3°.  $[\alpha]_D - 46.72^\circ$  in EtOH.

*l-Menthyl ester*: m.p. 64°.  $[\alpha]_D - 64.00^\circ$  in  $CHCl_3$ .

*Di-l-menthyl ester*:  $C_{24}H_{42}O_4$ . MW, 394. M.p. 63°.  $[\alpha]_D^{20} - 82.4^\circ$  in  $CHCl_3$ .

*l-Bornyl ester*:  $C_{14}H_{22}O_4$ . MW, 254. M.p. 58°.  $[\alpha]_D - 35.94^\circ$  in EtOH.

*Di-l-bornyl ester*:  $C_{24}H_{38}O_4$ . MW, 390. M.p. 83–7°.  $[\alpha]_D - 42.39^\circ$  in EtOH.

*Phenyl ester*:  $C_{10}H_{10}O_4$ . MW, 194. M.p. 98°.

*Diphenyl ester*:  $C_{16}H_{14}O_4$ . MW, 270. M.p. 121° (118°). B.p. 222.5°/15 mm.

*Di-o-nitrophenyl ester*:  $C_{16}H_{12}O_8N_2$ . MW, 360. M.p. 162°.

*Di-m-nitrophenyl ester*: m.p. 153°.

*Di-p-nitrophenyl ester*: m.p. 178°.

*Di-o-tolyl ester*:  $C_{18}H_{18}O_4$ . MW, 298. B.p. 238–40°/5 mm.

*Di-m-tolyl ester*: m.p. 60°.

*Di-p-tolyl ester*: m.p. 121°.

*Di-l-naphthyl ester*:  $C_{24}H_{18}O_4$ . MW, 370. M.p. 155°.

*Di-2-naphthyl ester*: m.p. 163°.

*Benzyl ester*:  $C_{11}H_{12}O_4$ . MW, 208. M.p. 46.5–47° (59°).

*Dibenzyl ester*:  $C_{18}H_{18}O_4$ . MW, 298. M.p. 45° (49–50°). B.p. 238°/14 mm.

*Amide*: see Succinamic Acid.

*Diamide*: see Succinamide.

*Imide*: see Succinimide.

*Chloride*: see Succinyl chloride.

*Bromide*: see Succinyl bromide.

*Anhydride*: see Succinic Anhydride.

*Nitrile*: see 2-Cyanopropionic Acid.

*Dinitrile*: see Succinonitrile.

*Et ester-nitrile*: see under 2-Cyanopropionic acid.

*Amide-nitrile*: see under 2-Cyanopropionic acid.

*Anilide*: see Succinanilic Acid.

*Dixanilide*: see Succinanilide.

*Di-2:4-dichloroanilide*: m.p. 248°.

*Mono-o-nitroanilide*: m.p. 132–132.5°.

*Mono-m-nitroanilide*: m.p. 181–2°.

*Mono-p-nitroanilide*: m.p. 196–7°.

*Di-p-nitroanilide*: m.p. 260°.

*Me-anilide*: m.p. 91–92.5°.

*Di-Me-anilide*: m.p. 156–5°.

*Et-anilide*: m.p. 92–3°.

*Di-Et-anilide*: m.p. 106°.

*o-Toluidide*: m.p. 97°.

*Di-o-toluidide*: m.p. 100°.

*p-Toluidide*: m.p. 179–80°.

*Di-p-toluidide*: m.p. 256°.

*1-Naphthalide*: m.p. 171°.

*Di-l-naphthalide*: m.p. 285° decomp.

*2-Naphthalide*: m.p. 190–2°. *Et ester*: m.p. 99–100°.

*Di-2-naphthalide*: m.p. 266°.

*Peroxide*: see Succinperoxide.

Leffler, Adams, *J. Am. Chem. Soc.*, 1936, **58**, 1553.

I.G., D.R.P., 485,313, (*Chem. Abstracts*, 1930, **24**, 862).

Fourneau, Sabetay, *Bull. soc. chim.*, 1929, **45**, 834.

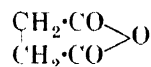
Zaidan, Kenkyuja, D.R.P., 469,234, (*Chem. Abstracts*, 1929, **23**, 2110).

Conzen-Crowet, *Bull. soc. chim. Belg.*, 1926, **35**, 165.

### Succinic Aldehyde.

See Succindialdehyde.

**Succinic Anhydride** (2:5-Diketotetrahydrofuran)



$C_4H_4O_3$  MW, 100

Cryst. from  $CHCl_3$ . M.p. 119.3–119.6°. B.p. 261°, 189°/100 mm., 169°/50 mm., 139°/15 mm., 131°/10 mm. Sol.  $CHCl_3$ . Spar. sol.  $Et_2O$ .  $D_4^{20}$  1.2340. Heat of comb.  $C_p$  373.1 Cal.

Leffler, Adams, *J. Am. Chem. Soc.*, 1936, **58**, 1551.

Jeffery, Vogel, *J. Chem. Soc.*, 1934, 1103  
Shriner, Struck, *Organic Syntheses*, 1932  
XII, 66.

Jaeger, Fiedler, U.S.P., 1,929,381, (*Chem. Abstracts*, 1934, **28**, 180).

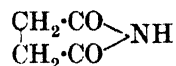
### Succinic Dialdehyde.

See Succindialdehyde.

### Succinic Semialdehyde.

See 2-Aldehydopropionic Acid.

**Succinimide** (2:5-Diketopyrrolidine).



$C_4H_5O_2N$  MW, 99

Plates +  $1H_2O$  from EtOH. M.p. 126–7° (125–6°). B.p. 287–8° decomp. Spar. sol.  $Et_2O$ .  $k = 3 \times 10^{-11}$  at 25°. Forms N-metallic derivs.

*Oxime*: m.p. 197° decomp. *Benzoyl deriv.*: m.p. 184°.

*Dioxime*: m.p. 207°. *Diacetyl deriv.*: m.p. 170–1°. *Dibenzoyl deriv.*: m.p. 187°.

*N-Acetyl*: b.p. 167°/9.5 mm.

*N-Benzoyl*: m.p. 129–30°.

*N-Me*: see *N-Methylsuccinimide*.

*N-Et*:  $C_6H_9O_2N$ . MW, 127. M.p. 26°. B.p. 236°.

*N-2-Bromoethyl*:  $C_6H_8O_2NBr$ . 206. M.p. 56–7°.

*N-Propyl*:  $C_7H_{11}O_2N$ . MW, 141. M.p. 15–16°. B.p. 247–8°/763 mm., 136–7°/27 mm.

*N-3-Bromopropyl*:  $C_7H_{10}O_2NBr$ . MW, 220. M.p. 52°.

*N-Isopropyl*: m.p. 61°. B.p. 230°/755 mm., 225°/743 mm.

*N-sec.-n.-Butyl*:  $C_8H_{13}O_2N$ . MW, 155. B.p. 339–40°/758 mm.

*N-Isobutyl*: m.p. 28°. B.p. 247–8°/758 mm.

*N-Isoamyl*:  $C_9H_{15}O_2N$ . MW, 169. B.p. 261–2°.

*N-Allyl*:  $C_7H_9O_2N$ . MW, 139. B.p. 249–50°, 244–5°/730 mm., 130–1°/14 mm.

*N-Phenyl*: see *Succinil*.

*N-o-Nitrophenyl*:  $C_{10}H_8O_4N_2$ . MW, 220. M.p. 156°.

*N-m-Nitrophenyl*: m.p. 175–6° (172°).

*N-p-Nitrophenyl*: m.p. 208° (203–4°).

*N-p-Hydroxyphenyl*:  $C_{10}H_9O_3N$ . MW, 191. M.p. 275–6°. *Me ether*:  $C_{11}H_{11}O_3N$ . MW, 205.

M.p. 165°. *Et ether*: pyrantin.  $C_{12}H_{13}O_3N$ . MW, 219. M.p. 158° (155°). *Propyl ether*:  $C_{13}H_{15}O_3N$ . MW, 233. M.p. 178°.

*N-Benzoyl*: m.p. 215°.

*N-o-Tolyl*:  $C_{11}H_{11}O_2N$ . MW, 189. M.p. 75° (101–2°). B.p. 338–40°/733 mm. (339–40°/756 mm.).

*N-m-Tolyl*: m.p. 111–12°. B.p. 340–4°.

*N-p-Tolyl*: m.p. 151°. B.p. 344–5°/733 mm., 212°/13 mm.

*N-1-Naphthyl*:  $C_{14}H_{11}O_2N$ . MW, 225. M.p. 153°.

*N-2-Naphthyl*: m.p. 183°.

*N-Benzyl*:  $C_{11}H_{11}O_2N$ . MW, 189. M.p. 98–9° (103°).

*N-o-Nitrobenzyl*:  $C_{11}H_{10}O_4N$ . MW, 220. M.p. 130°.

*N-p-Nitrobenzyl*: m.p. 150–2°.

*N-Chloro*: cryst. from  $CCl_4$ . M.p. 150°.

*N-Bromo*: cryst. from  $C_6H_6$ . M.p. 173.5° decomp.

Roeder, *Ber.*, 1913, **46**, 2563.

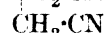
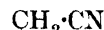
Ma, *Sah, Chem. Abstracts*, 1934, **28**, 6108.

Fehling, *Ann.*, 1844, **49**, 198.

Tscherniac, *Ber.*, 1901, **34**, 4213.

Swarts, *Am. Chem. J.*, 1897, **19**, 297.

**Succinonitrile** (*Ethylene dicyanide, succinic acid dinitrile, sym.-dicyanoethane*)



MW, 80

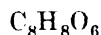
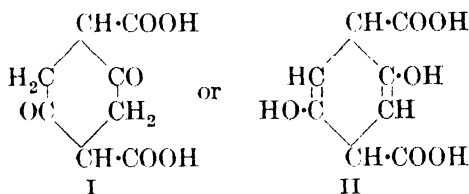
F.p. 53°. M.p. 53.7° (54.5°). B.p. 265–7°, 185°/60 mm., 158–60°/20 mm. Sol.  $H_2O$ ,  $EtOH$ ,  $CHCl_3$ . Spar. sol.  $Et_2O$ ,  $CS_2$ .

Fauconnier, *Bull. soc. chim.*, 1888, **50**, 214.

### Succinophenone.

See *Diphenacyl*.

**Succinosuccinic Acid** (2:5-Diketocyclohexane-1:4-dicarboxylic acid, 2:5-diketohexahydrotetraphthalic acid, succinylsuccinic acid, 1:4-dihydroxy- $\Delta^{1,4}$ -cyclohexadiene-3:6-dicarboxylic acid).



MW, 200

Needles. Spar. sol.  $H_2O$ .  $FeCl_3 \rightarrow$  violet col. in  $EtOH$ .

*Di-Me ester*:  $C_{10}H_{12}O_6$ . MW, 228. Needles from  $EtOH$ . M.p. 153°.

*Et ester*:  $C_{10}H_{12}O_6$ . MW, 228. M.p. 98° decomp.

*Di-Et ester*:  $C_{12}H_{16}O_6$ . MW, 256. (I). M.p. 123°. (II). M.p. 127°.

*Propyl ester*:  $C_{11}H_{14}O_6$ . MW, 242. Yellow needles from  $C_6H_6$ . M.p. 115° decomp.

*Dipropyl ester*:  $C_{14}H_{20}O_6$ . MW, 284. M.p. 91°.

*Isobutyl ester*:  $C_{12}H_{16}O_6$ . MW, 256. M.p. 126° decomp.

*Di-isobutyl ester*:  $C_{16}H_{24}O_6$ . MW, 312. M.p. 100°.

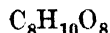
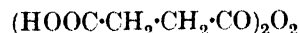
*Diallyl ester*:  $C_{14}H_{10}O_6$ . MW, 274. M.p. 115°.

Liebermann, *Ann.*, 1914, **404**, 287.

Baeyer, Noyes, *Ber.*, 1889, **22**, 2168.

Hantzsch, *Ber.*, 1915, **48**, 772.

### Succinperoxide (*Succinic acid peroxide*)



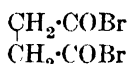
MW, 234

Plates. M.p. 128° decomp. Sol. H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin.

Clover, Houghton, *Am. Chem. J.*, 1904, 32, 55.

Stearns, D.R.P., 170,727, (*Chem. Zentr.*, 1906, II, 79).

### Succinyl bromide

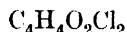
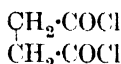


MW, 244

B.p. 105–6°/13 mm.

Hughes, Watson, *J. Chem. Soc.*, 1930, 1735.

### Succinyl chloride



MW, 155

Leaflets. M.p. 20° (17°). B.p. 190–2° (193·3°), 150–2°/214 mm., 103–4°/25 mm., 88·5°/19 mm.  $D_4^{25}$  1·3948.  $n_D^{25}$  1·473.

Vorländer, *Ann.*, 1894, 280, 183.

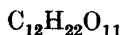
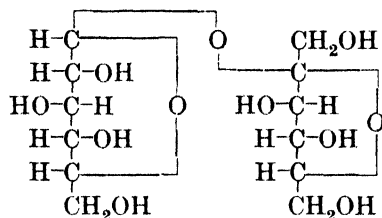
Morrell, *J. Chem. Soc.*, 1914, 105, 1733.

Monsanto, E.P., 418,162, (*Chem. Abstracts*, 1935, 29, 1436).

### Sucrol.

See Dulcin.

**Sucrose** (*Saccharose*, *cane sugar*)



MW, 342

Occurs in ripe fruits, sugar cane, beet, etc. Cryst. M.p. 184–5° from dil. EtOH, 179–80° (174°) from EtOH, 169–70° from MeOH. Sweet taste. 100 parts H<sub>2</sub>O dissolve 198·6 parts at 12·5°, 245·0 parts at 45°. Sol. MeOH. Spar. sol. 95% EtOH. Insol. Et<sub>2</sub>O.  $[\alpha]_D^{20} + 66·37^\circ$  in H<sub>2</sub>O.  $D^{17·5}$  1·5805,  $D^{30}$  1·5737 from MeOH, 1·5840 from EtOH. Does not reduce Fehling's. Does not form an osazone. Hyd. by dil. acids or by invertase  $\rightarrow$  glucose + fructose. Mod. stable to alkalis. Fermentable.  $\text{KMnO}_4 \rightarrow$  oxalic acid + CO<sub>2</sub>.  $\text{HNO}_3 \rightarrow$  saccharic, tartaric, and oxalic acids. Heat of

comb.  $C_v = 3945·7$  Cal. Forms compounds with CaO, BaO and SrO and with halides of alkali metals.

*Octa-Me ether*: syrup. B.p. 176°/0·05 mm.  $[\alpha]_D^{20} + 69·3^\circ$  in MeOH, + 66·8° in Me<sub>2</sub>CO.  $D_4^{20}$  1·1406.  $n_D$  1·4588. Hyd.  $\rightarrow$  2 : 3 : 4 : 6-tetramethylglucose + 1 : 3 : 4 : 6-tetramethyl- $\gamma$ -fructose.

*Octapropyl ether*: m.p. 45·4–45·5°.

*Octanitrate*: needles. M.p. 85·5°. Decomp. at 135°.  $[\alpha]_D^{20} + 55·9^\circ$  in MeOH. Sol. MeOH, Et<sub>2</sub>O. Spar. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, pet. ether.

*Octa-acetyl*: needles from EtOH. M.p. 72·3° (69°, 70°).  $[\alpha]_D^{20} + 59·6^\circ$  in CHCl<sub>3</sub>. Sol. hot EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Prac. insol. hot H<sub>2</sub>O. Insol. pet. ether.

*Hexabenzoyl*: cryst. M.p. 109°.

*Heptabenzoyl*: cryst. M.p. 98°.

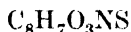
Avery, Haworth, Hirst, *J. Chem. Soc.*, 1927, 2308.

Josephson, *Ann.*, 1929, 472, 237.

### Sulphanilic Acid.

See Aniline-*p*-sulphonic Acid.

### Sulphazone



MW, 197

Brown leaflets from EtOH.Aq. M.p. 207–8°. Sol. EtOH, Me<sub>2</sub>CO, AcOH. Spar. sol. Et<sub>2</sub>O, hot H<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin.

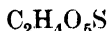
*B, HNO<sub>3</sub>*: yellow leaflets. Explodes at 172°.

*Me ether*: C<sub>9</sub>H<sub>9</sub>O<sub>3</sub>NS. MW, 211. Leaflets from MeOH.Aq. M.p. 210°.

Claasz, *Ber.*, 1916, 49, 614.

M.L.B., D.R.P., 269,428, (*Chem. Abstracts*, 1914, 8, 2035).

### Sulphoacetic Acid



MW, 140

Hygroscopic cryst. + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 84–6° (about 75°). Mod. sol. EtOH, Me<sub>2</sub>CO. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>.

*C-Anilide*: *Na salt*, m.p. 284°. *Aniline salt*, m.p. 229–31°.

*C-o-Toluidide*: *Na salt*, m.p. 223–4°. *o-Toluidine salt*: m.p. 189–91°.

*C-p-Anisidide*: *p-anisidine salt*, m.p. 224–7°.

*C-p-Phenetidide*: *Na salt*, m.p. 270°.

*p-Phenetidine salt*: m.p. 233–40°.

C-1-Naphthalide : Na salt, m.p. 285°.

Stillich, *J. prakt. Chem.*, 1906, **73**, 538;  
1906, **74**, 53.

Fichter, *Z. Elektrochem.*, 1914, **20**, 471.

Lichtenhahn, *Ber.*, 1915, **48**, 1950.

### Sulphoalanine.

See Cysteic Acid and Isocysteic Acid.

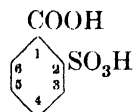
### Sulphoanistic Acid.

See under 4-Hydroxysulphobenzoic Acid.

### Sulphobenzide.

See Diphenyl sulphone.

**o-Sulphobenzoic Acid** (*Benzoic acid o-sulphonic acid*)



$C_7H_6O_5S$

MW, 202

Needles :  $3H_2O$  from  $H_2O$ . M.p. 68–9°, anhyd. 134° (141°). Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ . Heat  $\rightarrow$  anhydride.

1-Me ester : chloride.  $C_8H_7O_4ClS$ . MW, 234.5. M.p. 64–5°. Sol. EtOH,  $Et_2O$ . Amide :  $C_8H_5O_4NS$ . MW, 215. M.p. 125–6°.

1-Et ester : amide.  $C_9H_{11}O_4NS$ . MW, 229. M.p. 84°.

Di-Et ester :  $C_{11}H_{14}O_5S$ . MW, 258. B.p. 212–13°/21 mm.

1-Phenyl ester :  $C_{13}H_{10}O_5S$ . MW, 278. Needles from  $H_2O$ . M.p. 277–80°. Sol.  $H_2O$ , AcOH. Spar. sol. EtOH. Chloride :  $C_{13}H_9O_4Cl$ . MW, 264.5. Prisms from AcOH. M.p. 103–4°. Sol. EtOH, AcOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ . Amide :  $C_{13}H_{11}O_4NS$ . MW, 277. Prisms. M.p. 132°.

2-Phenyl ester : amide. M.p. 95°.

Diphenyl ester :  $C_{19}H_{14}O_5S$ . MW, 354. Needles from AcOH. M.p. 117.5–118.5°.

1-o-Tolyl ester : chloride.  $C_{14}H_{11}O_4Cl$ . MW, 278.5. M.p. 112°. Amide :  $C_{14}H_{13}O_4NS$ . MW, 291. M.p. 152°.

2-Chloride : nitrile.  $C_7H_4O_2NCIS$ . MW, 201.5. Prisms from  $Et_2O$ . M.p. 69–70° (67.5°). Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , hot pet. ether.

Dichloride :  $C_7H_4O_3Cl_2S$ . MW, 239. (i) Needles from pet. ether. M.p. 79°. Sol.  $Et_2O$ ,  $CHCl_3$ . Spar. sol. pet. ether. (ii) Plates from  $Et_2O$ . M.p. 40°. Sol.  $Et_2O$ ,  $CHCl_3$ .

1-Amide :  $C_7H_7O_4NS$ . MW, 201. Prisms +  $1H_2O$ . M.p. anhyd. 185–6°, (193–4°). Sol.  $H_2O$ , EtOH.

2-Amide : plates from  $H_2O$ . M.p. 165–7° (rapid heat.), 153–5° (slow heat.). Sol.  $H_2O$ , EtOH,  $Et_2O$ .  $k = 2.06 \times 10^{-3}$  at 25°.  $K$  deriv. : m.p. 285–6°. o-Toluidide : m.p. 193°.

p-Toluidide : m.p. 202°. Hydrazide : m.p. 182°.

1-Methylamide :  $C_8H_9O_4NS$ . MW, 215. M.p. 126°.

1-Ethylamide :  $C_9H_{11}O_4NS$ . MW, 229. M.p. about 111°.

Imide : see Saccharin.

Anhydride :  $C_7H_4O_4S$ . MW, 184. Plates from  $C_6H_6$ . M.p. 129.5° (128°). B.p. 184–6°/18 mm. Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , hot  $H_2O$ .

Nitrile : o-cyanobenzenesulphonic acid.  $C_7H_5O_3NS$ . MW, 183. Needles from  $H_2O$ . M.p. 279–279.5°. Mod. sol. EtOH, hot  $H_2O$ . Spar. sol.  $Et_2O$ ,  $CHCl_3$ . Anilide : m.p. 150–2°.

2-Anilide : m.p. 156°.

Dianilide : m.p. 194–5°.

Maarse, *Rec. trav. chim.*, 1914, **33**, 210.

Krannich, *Ber.*, 1900, **33**, 3485.

White, Acree, *Organic Syntheses*, Collective Vol. I, 13, 482.

Auger, Vary, *Bull. soc. chim.*, 1921, **29**, 990.

Ray, Dey, *J. Chem. Soc.*, 1920, **117**, 1406.

**m-Sulphobenzoic Acid** (*Benzoic acid m-sulphonic acid*).

Cryst. +  $2H_2O$ . M.p. 98°, anhyd. 141°. Sol.  $H_2O$ , EtOH. Insol.  $C_6H_6$ ,  $CHCl_3$ , pet. ether. Anhyd. acid sol.  $Et_2O$ .

1-Me ester :  $C_8H_8O_5S$ . MW, 216. M.p. 65°. Chloride :  $C_8H_7O_4ClS$ . MW, 234.5. M.p. 63–5°.

3-Me ester : m.p. 139–40°.

Di-Me ester :  $C_9H_{10}O_5S$ . MW, 230. M.p. 32–3°. B.p. 198–200°/20 mm.

3-Chloride :  $C_7H_5O_4ClS$ . MW, 220.5. M.p. 133–4°.

Dichloride :  $C_7H_4O_3Cl_2S$ . MW, 239. M.p. 20–4°.

3-Amide :  $C_7H_7O_4NS$ . MW, 201. M.p. 237–8° (233°). Nitrile :  $C_7H_6O_2N_2S$ . MW, 182. M.p. 151–2°.

Diamide :  $C_7H_8O_3N_2S$ . MW, 200. M.p. anhyd. 170°.

Offermann, *Ann.*, 1894, **280**, 6.

Maarse, *Rec. trav. chim.*, 1914, **33**, 209.

Nakaseko, *Am. Chem. J.*, 1912, **47**, 448.

**p-Sulphobenzoic Acid** (*Benzoic acid p-sulphonic acid*).

Needles +  $3H_2O$ . M.p. 94°, anhyd. 259–60°. Sol.  $H_2O$ , EtOH. Anhyd. acid sol.  $Et_2O$ .

1-Me ester : m.p. 99–100°.

4-Me ester : m.p. 195–6°.

Di-Me ester : m.p. 88–90°.

1-*Et ester*: amide.  $C_9H_{11}O_4NS$ . MW, 229. M.p. 110–11°  $\rightarrow$  solid, m.p. 94–5°.

4-*Chloride*: nitrile.  $C_7H_4O_2NClS$ . MW, 201.5. M.p. 111–12°.

*Dichloride*: m.p. 56–7°.

4-*Amide*: decomp. at 280°.  $k = 2.56 \times 10^{-4}$  at 25°. N-*Dichloro*: see Halazone. *Nitrile*:  $C_7H_6O_2N_2S$ . MW, 182. M.p. 168–9°.

*Diamide*: m.p. 236°.

*Nitrile*: anilide. M.p. 112°. m-*Toluidide*: m.p. 128°.

*Dianilide*: m.p. 252–3° decomp.

*Di-m-toluidide*: m.p. 241–2° decomp.

Heinemann, E.P., 23.575, (*Chem. Abstracts*, 1916, 10, 1579).

Remsen, *Ann.*, 1875, 178, 288.

See also second reference above.

### Sulphocresotic Acid.

See Hydroxy-sulphotoluic Acid.

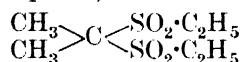
### Sulphoethylurea.

See Taurocarbamic Acid.

### 2-Sulphoethyl Alcohol.

See Isethionic Acid.

**Sulphonal** (*Acetone diethylsulphone, propane-2 : 2-diethyldisulphone*)



$C_7H_{16}O_4S_2$  MW, 228

Prisms from EtOH, microscopic leaflets from Et<sub>2</sub>O. M.p. 125.8°. B.p. 300° (slight decomp.). Sublimes (begins at 66°). Volatile in steam. Solubilities (1 gm. in  $x$  gms. solvent): H<sub>2</sub>O, 422 at 18°, 222 at 37°, 8 at 100°; EtOH, 2 at 78°; Et<sub>2</sub>O, 67 at 19°, 79 at 15°; CHCl<sub>3</sub>, 3.3 at 20°; C<sub>6</sub>H<sub>6</sub>, 12.7 at 20°; AcOEt, 13.7 at 20°; CCl<sub>4</sub>, 110 at 20°. Stable to acids, alkalis, bromine. Hypnotic.

Falck, *Chem. Abstracts*, 1920, 14, 1002.

Hirayama, Matsuzaki, Okamoto, *Chem. Abstracts*, 1918, 12, 1586.

**Sulphonyldiacetic Acid** (*Dimethylsulphone- $\alpha$  :  $\alpha'$ -dicarboxylic acid*)



$C_4H_6O_6S$  MW, 182

Plates. M.p. 182°. Decomp. at 200°  $\rightarrow$  dimethyl sulphone. Very sol. H<sub>2</sub>O, EtOH. Mod. sol. H<sub>2</sub>SO<sub>4</sub>. Aq., Et<sub>2</sub>O.  $k$  (first) =  $1.3 \times 10^{-2}$ ; (second) =  $4.75 \times 10^{-4}$  at 25°. Electrolysis of K salts  $\rightarrow$  H<sub>2</sub>SO<sub>4</sub> + CO<sub>2</sub>. NaNO<sub>2</sub>. Aq.  $\rightarrow$  CO<sub>2</sub> + HCN + H<sub>2</sub>SO<sub>4</sub>.

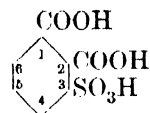
Fichter, Krummenacher, *Helv. Chim. Acta*, 1918, 1, 162.

Lovén, *Ber.*, 1884, 17, 2818.

### Sulphonyldiphenylmethane.

See Diphenylmethane sulphone.

**3-Sulphophthalic Acid** (*Phthalic acid 3-sulphonic acid*)



$C_8H_6O_7S$  MW, 246

Prisms + H<sub>2</sub>O of cryst. M.p. 62–4°. Very sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

3-*Amide*:  $C_8H_7O_6NS$ . MW, 245. Needles + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 155° decomp. (softens at 120°). *Di-Me ester*: m.p. 135°. *Di-Et ester*: m.p. 102°.

Remsen, Stokes, *Am. Chem. J.*, 1884, 6, 279.

Moulton, *Am. Chem. J.*, 1891, 13, 203.

Cf. Rée, *Ann.*, 1886, 233, 217.

**4-Sulphophthalic Acid** (*Phthalic acid 4-sulphonic acid*).

Cryst. + 1H<sub>2</sub>O. M.p. 138–40°. Very sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O. Fused with NaOH at 220°  $\rightarrow$  4-hydroxyphthalic acid. K salt heated with Na formate  $\rightarrow$  trimellitic acid.

4-*Chloride*:  $C_8H_5O_6ClS$ . MW, 264.5. Prisms from Et<sub>2</sub>O. M.p. 167–70° decomp.

4-*Amide*: prisms from H<sub>2</sub>O. M.p. 192–200° decomp.

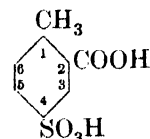
Bentley, Weizmann, *J. Chem. Soc.*, 1907, 91, 100.

Rée, *Ann.*, 1886, 233, 219, 228.

### Sulphosalicylic Acid.

See 2-Hydroxy-4-sulphobenzoic Acid and 6-Hydroxy-3-sulphobenzoic Acid.

### 4-Sulpho-o-toluic Acid



$C_8H_8O_5S$  MW, 216

Fibrous cryst. mass. Very sol. H<sub>2</sub>O. Fused with KOH  $\rightarrow$  4-hydroxy-o-toluic acid.

4-*Amide*:  $C_8H_9O_4NS$ . MW, 215. Needles. M.p. 243°.

Jacobsen, Wierss, *Ber.*, 1883, 16, 1959.

Baudisch, Perkin, *J. Chem. Soc.*, 1909, 95, 1883.

### 5-Sulpho-o-toluic Acid.

*Mono-NH<sub>4</sub> salt*: prisms from H<sub>2</sub>O. M.p. 284°. Reacts acid.

2-*Amide*:  $C_8H_9O_4NS$ . MW, 215. Needles

from  $\text{H}_2\text{O}$ . M.p.  $217^\circ$ .  $\text{NH}_4$  salt: prisms. M.p.  $276-8^\circ$ .

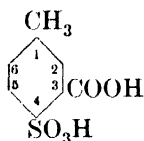
5-Amide: m.p.  $211^\circ$ .

Amide-nitrile:  $\text{C}_8\text{H}_8\text{O}_2\text{N}_2\text{S}$ . MW, 196. Cryst. M.p.  $160^\circ$ .

Chloride-nitrile:  $\text{C}_8\text{H}_6\text{O}_2\text{NClS}$ . MW, 215.5. Cryst. from ligroin. M.p.  $53^\circ$ .

Nowell, *Am. Chem. J.*, 1912, **48**, 225, 237.

#### 4-Sulpho-*m*-toluic Acid.



$\text{C}_8\text{H}_8\text{O}_5\text{S}$

MW, 216

Fused with  $\text{KOH} \rightarrow$  4-hydroxy-*m*-toluic acid.

Meldrum, Perkin, *J. Chem. Soc.*, 1909, **95**, 1891.

#### 5-Sulpho-*m*-toluic Acid.

Cryst. +  $2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $110^\circ$ . Fused with  $\text{KOH} \rightarrow$  5-hydroxy-*m*-toluic acid.

See previous reference.

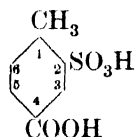
#### 6-Sulpho-*m*-toluic Acid.

6-Amide: needles from  $\text{H}_2\text{O}$ . M.p.  $254^\circ$  ( $220^\circ$  slow heat.).

Waters, *Am. Chem. J.*, 1912, **47**, 343, 349.

Meldrum, Perkin, *J. Chem. Soc.*, 1909, **95**, 1891.

#### 2-Sulpho-*p*-toluic Acid



$\text{C}_8\text{H}_8\text{O}_5\text{S}$

MW, 216

Cryst. +  $1\text{H}_2\text{O}$  from  $\text{AcOH}$ , +  $3\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .

2-Amide: needles from  $\text{H}_2\text{O}$ . M.p.  $267^\circ$ .

Diamide:  $\text{C}_8\text{H}_{10}\text{O}_3\text{N}_2\text{S}$ . MW, 214. Needles +  $\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $228^\circ$ .

Meldrum, Perkin, *J. Chem. Soc.*, 1908, **93**, 1419.

Weinreich, *Ber.*, 1887, **20**, 981.

#### 3-Sulpho-*p*-toluic Acid.

Cryst. +  $3\text{H}_2\text{O}$ . M.p. anhyd.  $190^\circ$  ( $158^\circ$ ). Sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Fused with  $\text{KOH} \rightarrow$  *m*-cresotic acid.

3-Amide: cryst. from  $\text{H}_2\text{O}$ . M.p.  $185^\circ$  ( $181^\circ$ ). *Me* ester: m.p.  $145^\circ$ . *Et* ester: m.p.  $95^\circ$ .

4-Amide:  $\text{NH}_4$  salt, cryst. +  $1\text{H}_2\text{O}$ . M.p.  $186^\circ$ .

Amide-nitrile: leaflets from  $\text{Py}$ . Very spar. sol.  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Sol. alkalis.

Dichloride:  $\text{C}_8\text{H}_6\text{O}_3\text{Cl}_2\text{S}$ . MW, 253. Cryst. from pet. ether. M.p.  $59^\circ$ .

Chloride-nitrile: leaflets from ligroin. M.p.  $67^\circ$ .

Weber, *Ber.*, 1892, **25**, 1741.

Randall, *Am. Chem. J.*, 1891, **13**, 258.

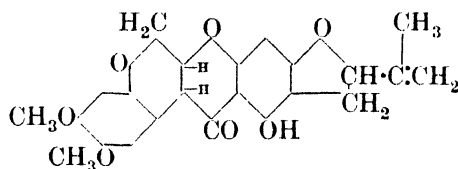
#### Sulphovinic Acid.

See Ethyl hydrogen sulphate.

#### Sulphuric Ether.

See Diethyl Ether.

#### Sumatrol



Suggested structure

$\text{C}_{23}\text{H}_{22}\text{O}_7$

MW, 410

Occurs in resin from *Derris* species. Needles from  $\text{EtOH}$ . M.p.  $195-6^\circ$  (air-dried). Recryst. from  $\text{Me}_2\text{CO}$ , m.p.  $183^\circ \rightarrow 194^\circ$  after several days keeping. Sol.  $\text{CHCl}_3$ . Mod. sol.  $\text{C}_6\text{H}_6$ ,  $\text{AcOEt}$ . Spar. sol.  $\text{MeOH}$ , cold  $\text{AcOH}$ , 8%  $\text{NaOH.Aq.}$   $[\alpha]_D -184^\circ$  in  $\text{C}_6\text{H}_6$ . Alc.  $\text{FeCl}_3 \rightarrow$  deep brownish-green col.

*Oxime*: needles from  $\text{EtOH.Aq.}$  M.p.  $245-7^\circ$ .

Robertson, Rusby, *J. Chem. Soc.*, 1937, **497**.

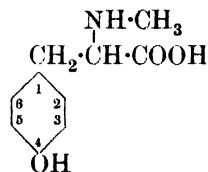
#### Superpalite.

See Trichloromethyl chloroformate.

#### Suprarenin.

See Adrenaline.

**Surinamine** (*Ratanhin*, *geoffroyin*, *angelin*, *andirin*, *N-methyltyrosine*)



$\text{C}_{10}\text{H}_{13}\text{O}_3\text{N}$

MW, 195

Occurs in bark of *Andira retusa*, H.B. and K. Needles. M.p.  $257^\circ$  (decomp. at  $280^\circ$ ).  $[\alpha]_D^{25} + 19.8^\circ$  in dil.  $\text{HCl}$ .

*Me* ester:  $\text{C}_{11}\text{H}_{15}\text{O}_3\text{N}$ . MW, 209. M.p.  $111-12^\circ$  ( $116-17^\circ$ ).

*Me ether-nitrile*:  $C_{11}H_{14}ON_2$ . MW, 190. M.p. 152–3°.

Fischer, Lipschitz, *Ber.*, 1915, **48**, 377.

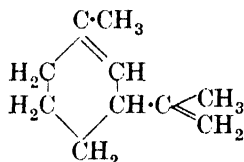
Winterstein, *Z. physiol. Chem.*, 1919, **105**, 20.

Kanevska, *J. prakt. Chem.*, 1929, **124**, 48.

### Sylvan.

See 2-Methylfuran.

**Sylvestrene** ( $\Delta^{1,8(9)}$ -*m*-Menthadiene, 1-methyl-3-isopropenylcyclohexene)



$C_{10}H_{16}$

MW, 136

Terpene hydrocarbon which does not occur in nature, but is generated from *carene* hydrocarbons during processes of isolation and purification of essential oils of *Pinus*. All forms sol.  $Ac_2O + 1$  drop conc.  $H_2SO_4 \rightarrow$  deep blue col.

*d*-.

B.p. 175°/751 mm. Polymerises on long heating.  $D^{18}_D$  0.8479.  $n^{18}_D$  1.4760.  $[\alpha]^{18}_D + 83.18^\circ$ . Heat of comb.  $C_r$  1464.2 Cal.

*Dihydrochloride*: m.p. 72°.

*Nitrosochloride*: m.p. 106–7°.

*l*-.

B.p. 176–8°.  $D^{19}_D$  0.848.  $n^{18}_D$  1.4761.  $[\alpha]^{18}_D - 68.2^\circ$  in  $AcOEt$ .

*Dihydrochloride*: m.p. 72°.

*dl*-. Carvestrene.

B.p. 178°. Resinifies in air.

*Dihydrochloride*: cryst. from  $MeOH$ . M.p. 52°.

Aschan, *Ann.*, 1928, **461**, 1.

Semmler, Schiller, *Ber.*, 1927, **60**, 1591.

Rao, Simonsen, *J. Chem. Soc.*, 1925, 2494.

Haworth, Perkin, Wallach, *J. Chem. Soc.*, 1913, **103**, 2233.

**Synthalin** (Decamethylenediguanidine, 1 : 10-diguanidino-*n*-decane)



$C_{12}H_{28}N_6$

MW, 256

Antidiabetic.

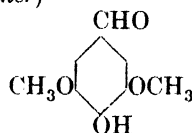
$B, 2HCl$ : cryst. from  $EtOH-Et_2O$ . M.p. 193°.

Kumagai et al., *Sci. Papers Inst. Phys. Chem. Res., Tokyo*, 1928, **9**, 271.

Schering-Kahlbaum A.G., E.P., 285,873, (*Chem. Abstracts*, 1929, **23**, 154).

Ammon, *Chem.-Tech. Rundschau*, 1930, **45**, 406 (Review).

**Syringa-aldehyde** (*Syringic aldehyde*, 4-hydroxy-3 : 5-dimethoxybenzaldehyde, gallaldehyde 3 : 5-dimethyl ether)



$C_9H_{10}O_4$

MW, 182

M.p. 113°. B.p. 192–3°/14 mm. Sol.  $EtOH$ ,  $Et_2O$ ,  $CHCl_3$ ,  $AcOH$ , hot  $C_6H_6$ .  $FeCl_3$ -Aq.  $\rightarrow$  olive-green col. The K and Na salts are yellow.

*O*-Carbomethoxyl: m.p. 98–9°.

*Hydrazone*: m.p. 208–9°.

*p*-Nitrophenylhydrazone: m.p. 216–17°.

*Semicarbazone*: m.p. 188°.

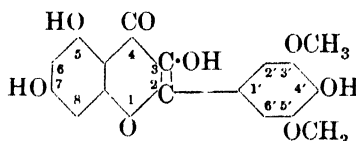
Sharp, *J. Chem. Soc.*, 1937, 853.

Mauthner, *J. prakt. Chem.*, 1935, **142**, 26.

McCord, *J. Am. Chem. Soc.*, 1931, **53**, 4181.

Pauly, Strassberger, *Ber.*, 1929, **62**, 2277. See also references under Gallaldehyde.

**Syringetin** (5 : 7 : 4'-Trihydroxy-3' : 5'-dimethoxyflavonol)



$C_{17}H_{14}O_8$

MW, 346

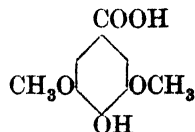
Pale yellow needles from  $AcOH$ . M.p. 288–9° (darkens at 270°). Mod. sol.  $MeOH$ ,  $EtOH$ ,  $Me_2CO$ ,  $AcOH$ . Spar. sol.  $AcOEt$ ,  $CHCl_3$ . Insol.  $C_6H_6$ , pet. ether. Conc.  $H_2SO_4 \rightarrow$  yellow col. with green fluor. Alk. sols. are intensely deep yellow.

*Tetra-acetyl*: m.p. 224–6°.

4'-Benzyl ether: m.p. 240–1°. *Triacetyl*: m.p. 191–4°.

Heap, Robinson, *J. Chem. Soc.*, 1929, 67.

**Syringic Acid** (4-Hydroxy-3 : 5-dimethoxybenzoic acid, gallic acid 3 : 5-dimethyl ether)



$C_9H_{10}O_5$

MW, 198

Cryst. M.p. 204–5°.

4-Acetyl: m.p. 191° (187°).





d.-

Laminæ from  $\text{Me}_2\text{CO}$ . M.p.  $158^\circ$  decomp. ( $155-8^\circ$ ).  $[\alpha]_D^{20} + 29.4^\circ$  in  $\text{H}_2\text{O}$  ( $[\alpha]_D + 29^\circ \rightarrow +6.7^\circ$  in  $\text{H}_2\text{O}$ ). Sol.  $\text{H}_2\text{O}$ , hot  $\text{EtOH}$ . Spar. sol.  $\text{Me}_2\text{CO}$ . Prac. insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Partly converted into mucic acid on boiling with  $\text{Py.Aq.}$

*Diphenylhydrazide*: m.p.  $185-90^\circ$ .

l.-

Cryst. from  $\text{Me}_2\text{CO}$ . M.p.  $158^\circ$  decomp.  $[\alpha]_D^{20} - 33.9^\circ$  in  $\text{H}_2\text{O}$ .

*Diphenylhydrazide*: m.p.  $185^\circ$ .

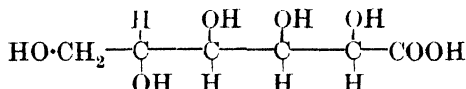
Fischer, *Ber.*, 1891, **24**, 3625.

Fischer, Morrell, *Ber.*, 1894, **27**, 391.

Levene, Jacobs, *Ber.*, 1910, **43**, 3145.

Steiger, Reichstein, *Helv. Chim. Acta*, 1936, **19**, 198.

### Talonic Acid


 $\text{C}_6\text{H}_{12}\text{O}_7$ 

MW, 196

d.-

Cryst. +  $\text{H}_2\text{O}$  from dil.  $\text{EtOH}$ . M.p.  $138-9^\circ$  ( $125^\circ$ ).  $[\alpha]_D^{25} + 16.73^\circ \rightarrow -21.57^\circ$  in  $\text{H}_2\text{O}$  ( $18.71^\circ$  in  $\text{H}_2\text{O}$ ). Sol. cold  $\text{H}_2\text{O}$ . Dil.  $\text{HNO}_3 \rightarrow$  talomucic acid.

*K salt*: m.p.  $171-2^\circ$  ( $169^\circ$ ).  $[\alpha]_D^{20} + 2.97^\circ$  in  $\text{H}_2\text{O}$  ( $[\alpha]_D^{20} + 3^\circ$  in  $\text{H}_2\text{O}$ ).

*NH<sub>4</sub> salt*: m.p.  $148^\circ$ .  $[\alpha]_D^{25} + 2.9^\circ$  in  $\text{H}_2\text{O}$ .

*Brucine salt*:  $\text{C}_{23}\text{H}_{26}\text{O}_4\text{N}_2 \cdot \text{C}_6\text{H}_{12}\text{O}_7$ . Needles +  $3\frac{1}{2}$  (?)  $\text{H}_2\text{O}$  from  $\text{EtOH}$ . M.p.  $95-100^\circ$ , anhyd.  $154-6^\circ$ .  $[\alpha]_D^{20} - 26.15^\circ$  in  $\text{H}_2\text{O}$ .

*$\gamma$ -Lactone*: m.p.  $135-7^\circ$  ( $132-4^\circ$ ).  $[\alpha]_D^{25} - 34.65^\circ \rightarrow -28.4^\circ$  in  $\text{H}_2\text{O}$ .  $\text{NaHg} \rightarrow d$ -talose.

*Amide*:  $\text{C}_6\text{H}_{13}\text{O}_6\text{N}$ . MW, 195. M.p.  $121^\circ$ .  $[\alpha]_D^{25} - 13.1^\circ \rightarrow 0^\circ$  in  $\text{EtOH}$ .

*Phenylhydrazide*: prisms from  $\text{EtOH}$ . M.p.  $161-2^\circ$  ( $159^\circ$ ).  $[\alpha]_D^{20} - 24.75^\circ \rightarrow 25.1^\circ$  in  $\text{H}_2\text{O}$  ( $[\alpha]_D^{25} - 25.43^\circ$  in  $\text{H}_2\text{O}$ ).

Hedenburg, Cretcher, *J. Am. Chem. Soc.*, 1927, **49**, 478.

Fischer, *Ber.*, 1891, **24**, 3622.

Fischer, Ruff, *Ber.*, 1900, **33**, 2146.

Cretcher, Renfrew, *J. Am. Chem. Soc.*, 1932, **54**, 1590, 4402.

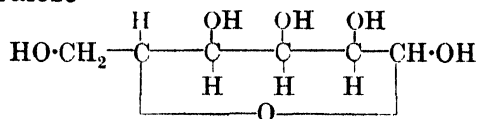
Brackenbury, Upson, *J. Am. Chem. Soc.*, 1933, **55**, 2512.

Bonnett, Upson, *ibid.*, 1247.

Bosshard, *Helv. Chim. Acta*, 1935, **18**, 485.

Steiger, Reichstein, *Helv. Chim. Acta*, 1936, **19**, 203.

### Talose


 $\text{C}_6\text{H}_{12}\text{O}_6$ 

MW, 180

d.-

M.p.  $128-30^\circ$ .  $[\alpha]_D^{21}$  about  $30^\circ \rightarrow 20.6^\circ$  in  $\text{H}_2\text{O}$  ( $[\alpha]_D^{27} + 78.7^\circ \rightarrow 46.3^\circ$  in  $\text{H}_2\text{O}$ ). Reduces Fehling's. Non-fermentable.

*Phenylhydrazone*: cryst. M.p.  $178^\circ$  decomp. *p*-Bromophenylhydrazone: cryst. from  $\text{EtOH}$ . M.p.  $205^\circ$ .

*Phenylosazone*: m.p.  $202^\circ$  decomp. ( $196-7^\circ$ ,  $188-91^\circ$ ). Identical with galactosazone.

*Methylphenylhydrazone*: cryst. from  $\text{MeOH}$ . M.p.  $220-2^\circ$  ( $154^\circ$ ).

*Benzylphenylhydrazone*: yellow laminæ. M.p.  $199^\circ$ .

*o*-Nitrophenylhydrazone: mp.  $148.5-149^\circ$ .  $[\alpha]_D^{18} + 88.3^\circ$  in  $\text{MeOH}$ .

v. Braun, Bayer, *Ber.*, 1925, **58**, 2221.

Blanksma, v. Ekenstein, *Chem. Zentr.*, 1908, II, 1584.

Bosshard, *Helv. Chim. Acta*, 1935, **18**, 482.

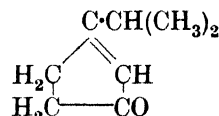
Komada, *Chem. Abstracts*, 1932, **26**, 4799.

Levene, *J. Biol. Chem.*, 1931, **93**, 631.

### Tanacetone.

See under Thujone.

**Tanacetophorone** (1-Isopropylcyclopentanone-3)


 $\text{C}_8\text{H}_{12}\text{O}$ 

MW, 124

Yellowish-brown oil. B.p.  $215-17^\circ$ ,  $83.5-84.5^\circ/11\text{ mm}$ .  $D_{20}^{20} 0.9378$ .  $n_D^{20} 1.4788$ . Volatile in steam. Red.  $\rightarrow$  3-isopropylcyclopentanone.

*Semicarbazone*: plates from  $\text{EtOH}$ . M.p.  $187-8^\circ$ .

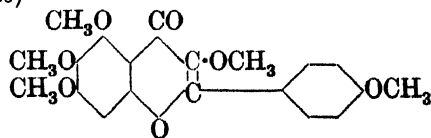
Pringsheim, Bondi, *Ber.*, 1925, **58**, 1415.

Wallach, *Ann.*, 1918, **414**, 221.

### Tanacetyl Alcohol.

See Thujyl Alcohol.

**Tangeritin** (3 : 5 : 6 : 7 : 4' - Pentamethoxy-flavone)


 $\text{C}_{20}\text{H}_{20}\text{O}_7$ 

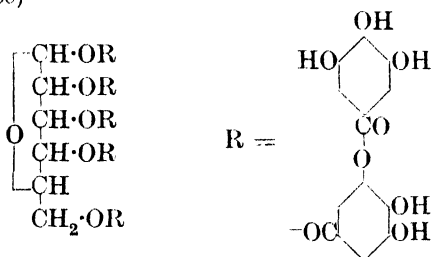
MW, 372

Constituent of peel of tangerines. Rods or needles from AcOEt. M.p. 154°. Sol. hot EtOH, hot AcOEt, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether. Insol. alkalis. Warm conc. HNO<sub>3</sub> → blood-red col. Hyd. → tangeretol + anisic acid. Boiling HI + phenol → 3 : 5 : 6 : 7 : 4'-penta-hydroxyflavone.

Robinson, Goldsworthy, *J. Chem. Soc.*, 1937, 46.

Nelson, *J. Am. Chem. Soc.*, 1934, 56, 1392.

**Tannic Acid** (*Tannin, gallotannin, Chinese tannin, gallotannic acid, penta-(m-digalloyl)-glucose*)



Probable constitution

C<sub>76</sub>H<sub>52</sub>O<sub>46</sub>

MW, 1700

Constituent of galls from many species of oak, particularly *Quercus lusitanica*, also in galls from *Rhus semilata*. Commercial product is insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. AcOEt. Sol. H<sub>2</sub>O, EtOH. Aq. sol. has astringent taste. FeCl<sub>3</sub> → bluish-black col. or ppt. Gives ppts. with most metallic salts, also with many alkaloids, albumen, gelatin, pyridine and quinoline acetate. Optically active with variable dextrorotation. Active principle in several "tannins" used in leather tanning. Has many industrial applications, e.g., as a mordant in the textile industries.

Russell, *Chemical Reviews*, 1935, 17, 160.

Takino, *Chem. Abstracts*, 1929, 23, 2707.

Hepworth, *J. Soc. Chem. Ind.*, 1923, 42, 41T.

Fischer, Bergmann, *Ber.*, 1919, 52, 829.

### Taraxanthin

C<sub>40</sub>H<sub>56</sub>O<sub>4</sub>

MW, 600

Isomeric with violaxanthin. Occurs as ester in flowers of dandelion (*Taraxacum officinale*, Wiggers), yellow coltsfoot (*Tussilago farfara*), buttercup (*Ranunculus acer*), etc. Yellow prisms from MeOH. M.p. 184–5°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 200° in AcOEt.

Dict. of Org. Comp.—III.

Absorption bands in CS<sub>2</sub> at 501, 469 and 441 m $\mu$ .

Kuhn, Grundmann, *Ber.*, 1934, 67, 596.

Karrer, Morf, *Helv. Chim. Acta*, 1932, 15, 863.

Kuhn, Lederer, *Z. physiol. Chem.*, 1931, 200, 108.

### Tariric Acid (5-Heptadecine-1-carboxylic acid)

CH<sub>3</sub>·[CH<sub>2</sub>]<sub>10</sub>·C:C·[CH<sub>2</sub>]<sub>4</sub>·COOH

C<sub>18</sub>H<sub>32</sub>O<sub>2</sub>

MW, 280

Occurs as glyceride in fruit of *Picramnia-Arten* (Tariri). Cryst. from EtOH. M.p. 50–5°. Red. → stearic acid. Ox. → lauric and glutaric acids. Br in cold CHCl<sub>3</sub> → dibromide. I in AcOH → di-iodide. Gives insol. Ag salt.

*Dibromide*: cryst. mass. M.p. 32°.

*Di-iodide*: needles from EtOH. M.p. 48–5°.

*Dichloride*: brown needles. M.p. about 28°.

*Tetrabromide*: see 5 : 5 : 6 : 6-Tetrabromostearic Acid.

Arnaud, Hasenfratz, *Compt. rend.*, 1911, 152, 1604.

Arnaud, Posternak, *Compt. rend.*, 1909, 149, 220.

### Taroxyllic Acid (5 : 6-Diketostearic acid)

CH<sub>3</sub>·[CH<sub>2</sub>]<sub>10</sub>·CO·CO·[CH<sub>2</sub>]<sub>4</sub>·COOH

C<sub>18</sub>H<sub>32</sub>O<sub>4</sub>

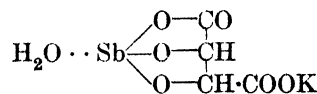
MW, 312

Pale yellow plates. M.p. 98°. Very sol. boiling EtOH. Spar. sol. cold EtOH. Insol. H<sub>2</sub>O. Alkali salts sol. H<sub>2</sub>O.

*Dioxime*: needles. M.p. 166–7°. Very sol. boiling EtOH. Insol. H<sub>2</sub>O.

Arnaud, *Compt. rend.*, 1902, 134, 548.

### Tartar emetic (Potassium antimonyl tartrate)



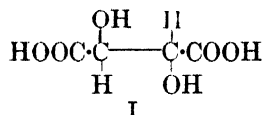
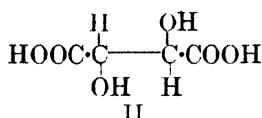
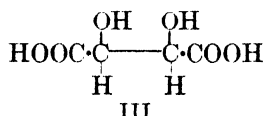
C<sub>4</sub>H<sub>4</sub>O<sub>7</sub>KSb

MW, 323

Rhombic cryst. D 2.607. Sol. 3 parts of H<sub>2</sub>O at 100°, 25 parts at 15°. One of the few soluble salts of antimony. Used in medicine in small doses: poisonous in larger quantities. Used for mordanting textiles in conjunction with tannic acid.

Reihlen, Hezel, *Ann.*, 1931, 487, 213.

Chemnitius, *Chem. Abstracts*, 1930, 24, 2832.

**Tartaric Acid** (*Dihydroxysuccinic acid*)*d*-*l**Meso*-*Racemic* = I + II. $\text{C}_4\text{H}_6\text{O}_6$ 

MW, 150

*d*-.  
Ordinary tartaric acid. Occurs in plants,

partly free and partly as K, Ca or Mg salts. The KH salt occurs in grape juice. Prisms. M.p. 170° (169°, 168–70°).  $D_4^{20}$  1.7594,  $D_4^{18}$  1.759,  $D_4^{20}$  1.7598. 100 Parts  $\text{H}_2\text{O}$  dissolve 115.04 parts at 0°, 139.44 parts at 20°, 147.44 parts at 25°, 195.0 parts at 50°, 258.05 parts at 75° and 343.35 parts at 100°. 1 Part dissolves in 2.06 parts 80% EtOH at 15°. 100 Parts 90% EtOH contain 29.146 parts at 15°. 100 Parts abs. EtOH contain 20.385 parts at 15°. 100 Parts  $\text{Et}_2\text{O}$  contain 0.393 parts at 15°. Sol.  $\text{Me}_2\text{CO}$ .  $[\alpha]_D^{20} + 11.98^\circ$  (20% sol. in  $\text{H}_2\text{O}$ ),  $[\alpha]_D^{16} + 13.1^\circ$  (15% sol. in  $\text{H}_2\text{O}$ ),  $[\alpha]_D + 0.47^\circ$  in MeOH. Heat of comb.  $\text{C}_p$  261.75 Cal.  $k$  (first) =  $1.3 \times 10^{-3}$  at 25° ( $1.02 \times 10^{-3}$ ,  $0.97 \times 10^{-3}$ ); (second) =  $69 \times 10^{-6}$  at 25° ( $97 \times 10^{-6}$ ,  $41 \times 10^{-6}$ ,  $45 \times 10^{-6}$ ,  $34.3 \times 10^{-6}$ ). Heat  $\rightarrow$  chars with evolution of  $\text{CO}_2$  and CO, and odour of burnt sugar (other products include acetaldehyde, acetone, acetic, formic, and pyruvic acids). On boiling with HCl.Aq. or dil.  $\text{H}_2\text{SO}_4$  or 30% NaOH it is converted into racemic and meso-tartaric acids.  $\text{H}_2\text{SO}_4$  at 100°  $\rightarrow$   $\text{CO}_2$ , CO and  $\text{SO}_2$ .  $\text{H}_2\text{O}_2$  (+ ferrous salt)  $\rightarrow$  dihydroxymaleic acid (Fenton's reaction).  $\text{MnO}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{H}\cdot\text{COOH} + \text{CO}_2$ . Reacts with cold  $\text{KMnO}_4$  with difficulty, but rapidly on warming. Reduces  $\text{NH}_3\cdot\text{AgNO}_3$ .  $\text{HI} \rightarrow d$ -malic acid + succinic acid.  $\text{HNO}_3 + \text{H}_2\text{SO}_4 \rightarrow$  di-nitrate of tartaric acid. Prevents the pptn. of CuO and other metallic oxides from alk. sol. Gives yellow col. with  $\text{FeCl}_3$ . Addn. of aq.  $\text{CaCl}_2$  to neutral sol. pptes. Ca salt, sol. AcOH and cold alkalis.

*Mono-NH<sub>4</sub> salt*: cryst. Triboluminescent. Sol. 45.6 parts  $\text{H}_2\text{O}$  at 15°.  $[\alpha]_D^{15} + 25.55^\circ$  in  $\text{H}_2\text{O}$ .

*Di-NH<sub>4</sub> salt*: cryst. Triboluminescent.  $[\alpha]_D^{15} + 34.6^\circ$  in  $\text{H}_2\text{O}$ . Spar. sol. EtOH.

*Mono-Na salt*: cryst. +  $\text{H}_2\text{O}$ . Triboluminescent.  $[\alpha]_D^{19} + 21.8^\circ$  in  $\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ .

*Di-Na salt*: cryst. +  $2\text{H}_2\text{O}$ . Triboluminescent. Sol. 3.46 parts  $\text{H}_2\text{O}$  at 6°, 2.28 parts at 24°, 1.5 parts at 42.5°.

*NaNH<sub>4</sub> salt*: cryst. +  $4\text{H}_2\text{O}$ . Triboluminescent. 100 parts aq. sol. at 0° contain 21.2 parts cryst. salt.

*Mono-K salt*: argol, tartar, cream of tartar (in order of increasing purity). Cryst. Triboluminescent. 100 c.cs. saturated aq. sol. contain 0.370 gm. at 0°, 0.843 gm. at 25°, 1.931 gm. at 50°, 5.850 gm. at 100°.

*Di-K salt*: cryst. +  $\frac{1}{2}\text{H}_2\text{O}$ . Triboluminescent.  $D_4^{20}$  1.984. Sol. 0.75 part  $\text{H}_2\text{O}$  at 2°, 0.66 part at 14°, 0.63 part at 23°, 0.47 part at 64°.

*KNH<sub>4</sub> salt*: cryst. +  $\frac{1}{2}\text{H}_2\text{O}$ . Readily sol.  $\text{H}_2\text{O}$ .

*KNa salt*: Rochelle Salt. Cryst. +  $4\text{H}_2\text{O}$ . Triboluminescent. Sol. 1.70 parts  $\text{H}_2\text{O}$  at 6°.

*Potassium antimonyl tartrate*: see Tartar emetic.

$\text{Ca}(\text{C}_4\text{H}_5\text{O}_6)_2$ : cryst. +  $2\text{H}_2\text{O}$ . 100 parts  $\text{H}_2\text{O}$  dissolve 0.710 part anhyd. salt at 15.6°. More sol. hot  $\text{H}_2\text{O}$ .

$\text{CaC}_4\text{H}_4\text{O}_6$ : cryst. +  $4\text{H}_2\text{O}$ . Occurs in many plants. 100 gm.  $\text{H}_2\text{O}$  dissolve 0.0185 gm. at 18°, 0.02948 gm. at 25° of hydrated salt. More sol. hot  $\text{H}_2\text{O}$ . Sol. hot alkalis.

$\text{AgC}_4\text{H}_5\text{O}_6$ : cryst. +  $\text{H}_2\text{O}$ . Sol. hot aq. tartaric acid.

$\text{Ag}_2\text{C}_4\text{H}_4\text{O}_6$ : cryst. powder. 100 gm.  $\text{H}_2\text{O}$  dissolve 0.2012 gm. at 18°, 0.2031 gm. at 25°. Insol. EtOH.

*Me ester*:  $\text{C}_5\text{H}_8\text{O}_6$ . MW, 164. Prisms +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. 76° (75–7°). 1 part dissolves in 4 parts  $\text{H}_2\text{O}$  at room temp. Sol. AcOEt,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{Et}_2\text{O}$ .  $[\alpha]_D^{18} + 18.71^\circ$  in  $\text{H}_2\text{O}$ .  $k = 4.6 \times 10^{-4}$  at 25°.

*Di-Me ester*: see Dimethyl tartrate.

*Et ester*:  $\text{C}_6\text{H}_{10}\text{O}_6$ . MW, 178. Prisms. M.p. about 90°. Deliquescent. Sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ .  $[\alpha]_D + 21.8^\circ$  in  $\text{H}_2\text{O}$ .

*Di-Et ester*: see Diethyl tartrate.

*Dipropyl ester*:  $\text{C}_{10}\text{H}_{18}\text{O}_6$ . MW, 234. B.p. 303°, 181°/23 mm., 171–2°/17 mm. (173–4°/17 mm.).  $D_4^{20}$  1.1344,  $D_4^{100}$  1.0590,  $D_4^{20}$  1.1390. Sol.  $\text{H}_2\text{O}$  and most org. solvents.  $[\alpha]_D^{20} + 12.44^\circ$  in  $\text{H}_2\text{O}$ . *Diacetyl*: m.p. 31°. B.p. 313°, 195–7°/13 mm.  $[\alpha]_D^{15} + 7.04^\circ$  in EtOH. *Dibenzoyl*:

m.p. 45.5°. B.p. 234°/7 mm.  $[\alpha]_{D}^{17.5} - 78.16^\circ$  in Py.

*Di-isopropyl ester*: b.p. 275°, 157–8°/16 mm.  $D^{20}_D$  1.1300,  $D^{100}_D$  1.0537.  $[\alpha]^{20}_D + 14.886^\circ$ . *Diacetyl*: prisms. M.p. 33°.  $[\alpha]^{20}_D + 5.9^\circ$  in EtOH.

*Dibutyl ester*:  $C_{12}H_{22}O_6$ . MW, 262. Prisms. M.p. 22–22.5°. B.p. 200–3°/18 mm., 178°/12 mm.  $D^{15}_D$  1.098.  $[\alpha]^{15}_D + 11.3^\circ$  in EtOH. *Diacetyl*: b.p. 214°/20 mm.  $D^{15.5}_D$  1.096.  $[\alpha]^{20}_D + 8.8^\circ$  in EtOH. *Dibenzoyl*: m.p. 43°. B.p. 250°/1.3 mm.  $[\alpha]^{15.5}_{461} - 57.62^\circ$  in Py.

*Di-isobutyl ester*: m.p. 68°. B.p. 323–5°, 197°/23 mm., 183°/11 mm., 157°/3.5 mm.  $D^{100}_D$  1.0145.  $[\alpha]_D + 11.8^\circ$  in EtOH. *Diacetyl*: b.p. 322–6°, 196–7°/12 mm.  $D^{15.5}_D$  1.096.  $[\alpha]^{14}_D + 10.51^\circ$  in EtOH. *Dibenzoyl*: b.p. about 240°/3 mm.  $D^{17.1}_D$  1.3360.  $[\alpha]^{12}_D - 48.86^\circ$  in EtOH.

*Di-d-amyl ester*:  $C_{14}H_{26}O_6$ . MW, 290. B.p. 208°/20 mm.  $D^{20}_D$  1.0636.  $[\alpha]^{20}_D + 17.73^\circ$ .

*Di-dl-amyl ester*: b.p. 208°/20 mm. (215–20°/10–25 mm.).  $D^{20}_D$  1.0637.  $[\alpha]^{20}_D + 14.10^\circ$ .

*Di-isoamyl ester*: b.p. 195°/16 mm.

*Diallyl ester*:  $C_{10}H_{14}O_6$ . MW, 230. B.p. 191°/20 mm., 171°/10 mm.

*Diphenyl ester*:  $C_{16}H_{14}O_6$ . MW, 302. Needles. M.p. 101–2°. Sol. hot EtOH, Et<sub>2</sub>O, glycerol. Insol. H<sub>2</sub>O.

*Dibenzyl ester*:  $C_{18}H_{18}O_6$ . MW, 330. M.p. about 50°. B.p. 250–70°/4 mm.  $D^{72.2}_D$  1.2036.

*Dibenzoyl*: needles from EtOH. M.p. 76–7°.  $[\alpha]^{18}_D + 6.2^\circ$  in Me<sub>2</sub>CO, + 41.7° in C<sub>6</sub>H<sub>6</sub>.

*Diphenacyl ester*:  $C_{20}H_{18}O_8$ . MW, 386. M.p. 130°.

*Anhydride*:  $C_4H_4O_5$ . MW, 132. *Dibenzoyl*: m.p. 173°.  $[\alpha]^{20}_D + 141.98^\circ$  in Me<sub>2</sub>CO.

*Amide*: see Tartramidic Acid.

*Diamide*: see under Tartramidic Acid.

*Dihydrazide*: needles. M.p. 182.5–183°. Sol. H<sub>2</sub>O. Spar. sol. EtOH. Insol. Et<sub>2</sub>O.  $[\alpha]^{20}_D + 97.1^\circ$  in H<sub>2</sub>O. B, 2HCl: cryst. Sol. H<sub>2</sub>O. Spar. sol. EtOH. Insol. Et<sub>2</sub>O. N : N-*Diacetyl*: needles from dil. EtOH. M.p. 216°. Sol. H<sub>2</sub>O, AcOH. Spar. sol. EtOH. Insol. Et<sub>2</sub>O.

*Di-azide*: cryst. M.p. 66° decomp. Sol. H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO. Mod. sol. Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin.

*Anilide*: see Tartranilic Acid.

*Dianilide*: see under Tartranilic Acid.

*Mono-Me ether*:  $C_8H_8O_6$ . MW, 164. Prisms from Et<sub>2</sub>O. M.p. 174°.  $[\alpha]_D + 45.4^\circ$  in H<sub>2</sub>O.

*Di-Me ether*: see Dimethoxysuccinic Acid.

*Di-Et ether*: see Diethoxysuccinic Acid.

*Diacetyl*: cryst. + 3H<sub>2</sub>O from Et<sub>2</sub>O. M.p. 58°. Sol. H<sub>2</sub>O, EtOH. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.  $[\alpha]^{22}_D - 23.04^\circ$  in H<sub>2</sub>O. Hyd. by warm aq. alkalis.

*Dibenzoyl*: cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 90° (88–9°), anhyd. 138–40°.  $[\alpha]^{18}_D - 115.78^\circ$  in EtOH,  $[\alpha]^{20}_D - 118.51^\circ$  in MeOH. Sol. EtOH. Less sol. CHCl<sub>3</sub>. Spar. sol. cold H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

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Rasch, D.R.P., 92,650, (*Chem. Zentr.*, 1897, II, 655).

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Gladys, D.R.P., 116,090, (*Chem. Zentr.*, 1901, I, 69).

McKenzie, *J. Chem. Soc.*, 1915, 107, 440.

Voss, *Chem.-Ztg.*, 1921, 45, 309, 335, 360, 411.

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Freundler, *Ann. chim. phys.*, 1894, 3, 445.

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*l.*

Physical properties identical with those of the *d*-acid.  $[\alpha]_D$  in H<sub>2</sub>O of same magnitude but opposite in sign to that of the *d*-acid.

*Di-Me ester*: see Dimethyl tartrate.

*Di-Et ester*: see Diethyl tartrate.

*Dibenzoyl*: cryst. + H<sub>2</sub>O. M.p. 85°.  $[\alpha]^{24}_D + 103.7^\circ$ .

*Di-Me ether*: see Dimethoxysuccinic Acid.

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Ladenburg, *Ann.*, 1909, 364, 232.

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Dale, Rice, *J. Am. Chem. Soc.*, 1933, 55, 4984.

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*dl.* Racemic Acid.

Cryst. anhyd. from H<sub>2</sub>O above 73° or from EtOH. M.p. 206° (205–6°). Cryst. + H<sub>2</sub>O, m.p. 203–4°. Loses H<sub>2</sub>O at 100°. Less sol. H<sub>2</sub>O than *d*- and *l*-acids. 100 Parts H<sub>2</sub>O dissolve 9.23 parts hydrated acid at 0°, 14.00 parts at 10°, 20.6 parts at 20°, 24.61 parts at 25°, 29.10 parts at 30°, 59.54 parts at 50°, 184.91 parts at 100°. Sol. 48 parts cold EtOH. Sol. in EtOH at 15° to extent of 2.08%, in Et<sub>2</sub>O 1.08%. Heat of comb. C<sub>r</sub> (hydrated) 278.4 Cal., (anhyd.) 279.5 Cal.  $k$  (first) =  $10.2 \times 10^{-4}$  at 25° ( $10.3 \times 10^{-4}$ ,  $11 \times 10^{-4}$ ,  $9.7 \times 10^{-4}$ ,  $9.6 \times 10^{-4}$ ); (second) =  $4.0 \times 10^{-5}$  at 25° ( $2.8 \times 10^{-5}$ ,  $3.94 \times 10^{-5}$ ). Heat with HCl.Aq. at 130–40° or in alk. sol. —> partly to mesotartaric acid.

*Mono-NH<sub>4</sub> salt*: prisms. Triboluminescent. Sol. 100 parts H<sub>2</sub>O at 20°. More sol. boiling H<sub>2</sub>O. Insol. EtOH.

*Di-NH<sub>4</sub> salt*: prisms. Sol. H<sub>2</sub>O. Prac. insol. EtOH.

*Mono-Na salt*: exists hydrated in several modifications. Loses  $\text{H}_2\text{O}$  at  $100^\circ$ . Triboluminescent. Decomp. at  $219^\circ$ . Sol. 11.3 parts  $\text{H}_2\text{O}$  at  $19^\circ$ . More sol. boiling  $\text{H}_2\text{O}$ . Insol. EtOH.

*Di-Na salt*: prisms. Triboluminescent. Sol. 2.63 parts  $\text{H}_2\text{O}$  at  $25^\circ$ . Insol. EtOH.

*$\text{NaNH}_4$  salt*: prisms +  $\text{H}_2\text{O}$ . Decomposes below  $27^\circ$  into d- and l- $\text{NaNH}_4$  tartrates, and above  $35^\circ$  into Na racemate and  $\text{NH}_4$  racemate.

*Mono-K salt*: prisms. Triboluminescent. Sol. 180 parts  $\text{H}_2\text{O}$  at  $19^\circ$ , 139 parts at  $25^\circ$ , 14.3 parts at  $100^\circ$ .

*Di-K salt*: plates +  $2\text{H}_2\text{O}$ . Sol. 0.97 part  $\text{H}_2\text{O}$  at  $25^\circ$ . Prac. insol. EtOH.

*KNa salt*: prisms +  $3\text{H}_2\text{O}$ . M.p.  $100^\circ$ . 100 gms. of saturated aq. sol. contains 36.66 gms. of anhyd. salt at  $9.7^\circ$ , 47.97 gms. at  $29.5^\circ$ . Also prisms or plates +  $4\text{H}_2\text{O}$ .

$\text{CaC}_4\text{H}_4\text{O}_6$ : cryst. +  $4\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  at  $200^\circ$ . Prac. insol. cold  $\text{H}_2\text{O}$ . More sol. hot  $\text{H}_2\text{O}$ . Insol. AcOH. Sol. HCl.Aq. but pptd. by  $\text{NH}_3$ .

*Me ester*: prisms. Sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Hyd. by boiling  $\text{H}_2\text{O}$ .

*Di-Me ester*: see Dimethyl tartrate.

*Et ester*: prisms. Sol.  $\text{H}_2\text{O}$ , EtOH. Insol.  $\text{Et}_2\text{O}$ .

*Di-Et ester*: see Diethyl tartrate.

*Dipropyl ester*: m.p.  $25^\circ$ . B.p.  $286^\circ/765$  mm.,  $167^\circ/11$  mm.  $D_4^{20}$  1.1256.

*Di-isopropyl ester*: m.p.  $34^\circ$ . B.p.  $275^\circ/765$  mm.,  $154^\circ/12$  mm.  $D_4^{20}$  1.1166.

*Dibutyl ester*: b.p.  $320^\circ/765$  mm.,  $185^\circ/12$  mm.  $D_4^{18}$  1.0879.

*Di-isobutyl ester*: m.p.  $58^\circ$ . B.p.  $311^\circ/768.5$  mm.,  $195^\circ/13$  mm.  $D_4^{20}$  1.0160.

*Di-d-amyl ester*:  $\text{C}_{14}\text{H}_{26}\text{O}_6$ . MW, 290. B.p.  $201.2^\circ/16$  mm. ( $215\text{--}25^\circ/10\text{--}25$  mm.).  $D_4^{20}$  1.064.  $n$  1.4501.  $[\alpha]_D^{20} + 3.37^\circ$ .

*Dibenzoyl*: m.p.  $112\text{--}13^\circ$ .

*Dinitrile*:  $\text{C}_4\text{H}_4\text{O}_2\text{N}_2$ . MW, 112. *Diacetyl*: plates or prisms from AcOH. M.p.  $97\text{--}8^\circ$ .

*Anhydride*: dibenzoyl, m.p.  $182^\circ$ .

*Di-Me ether*: see Dimethoxysuccinic Acid.

Campbell, Slotin, Johnston, *J. Am. Chem. Soc.*, 1933, 55, 2604.

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*Meso*-.

Plates +  $\text{H}_2\text{O}$ . M.p. anhyd.  $140^\circ$ .  $D_4^{20}$  1.666. Sol. 0.8 part  $\text{H}_2\text{O}$  at  $15^\circ$ .  $k$  (first) =  $6.0 \times 10^{-4}$

at  $25^\circ$  ( $6.3 \times 10^{-4}$ ); (second) =  $1.4 \times 10^{-5}$  at  $25^\circ$ . Heat with  $\text{H}_2\text{O}$  at  $175^\circ$ , with aq. HCl at  $130\text{--}40^\circ$  or with aq. alkalis  $\rightarrow$  partly to racemic acid.

*Mono-K salt*: needles. Sol. 8 parts  $\text{H}_2\text{O}$  at  $19^\circ$ .

$\text{CaC}_4\text{H}_4\text{O}_6$ : prisms +  $3\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  on standing, second  $\text{H}_2\text{O}$  at  $100^\circ$ , third  $\text{H}_2\text{O}$  at  $170^\circ$ . Sol. 600 parts boiling  $\text{H}_2\text{O}$ . Prac. insol. AcOH.

*Di-Me ester*: see under Dimethyl tartrate.

*Di-Et ester*: see under Diethyl tartrate.

*Dibutyl ester*: m.p.  $48\text{--}50^\circ$ .

*Di-d-amyl ester*: b.p.  $203\text{--}4^\circ/17$  mm.  $D_4^{20}$  1.0658.  $n_D^{20}$  1.4530.  $[\alpha]_D^{20} + 4.77^\circ$ .

*Dinitrile*: plates or prisms from  $\text{Et}_2\text{O}$ . M.p. about  $131^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{CHCl}_3$ . Prac. insol.  $\text{CS}_2$ . Not very stable. *Diacetyl*: prisms from  $\text{Et}_2\text{O}$  or AcOH. M.p.  $75\text{--}7^\circ$ .

*Anhydride*: dibenzoyl, m.p.  $207\text{--}8^\circ$ .

*Di-Me ether*: see Dimethoxysuccinic Acid.

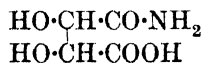
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Pollak, *Monatsh.*, 1894, 15, 469.

## Tartramide.

See under Tartramidic Acid.

## Tartramidic Acid (Tartaric monoamide)



$\text{C}_4\text{H}_7\text{O}_5\text{N}$

MW, 149

*d*-.

Cryst. from  $\text{H}_2\text{O}$ . M.p.  $171\text{--}2^\circ$ .  $[\alpha]_D^{15} + 63.7^\circ$  in  $\text{H}_2\text{O}$ .

$\text{Ca}(\text{C}_4\text{H}_6\text{O}_5\text{N})_2$ : cryst. +  $6\text{H}_2\text{O}$ . Sol.  $\text{H}_2\text{O}$ . Insol. EtOH.  $[\alpha]_D^{18} + 59.5^\circ$  in  $\text{H}_2\text{O}$ .

*Et ester*:  $\text{C}_6\text{H}_{11}\text{O}_5\text{N}$ . MW, 177. Plates. M.p.  $136\text{--}7^\circ$ .

*Amide*:  $\text{C}_4\text{H}_8\text{O}_4\text{N}_2$ . MW, 148. *d*-Tartramide, diamide of tartaric acid. Cryst. M.p.  $195^\circ$  decomp. Spar. sol. MeOH, EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .  $[\alpha]_D^{20} + 106.5^\circ$  in  $\text{H}_2\text{O}$ ,  $+144^\circ$  in MeOH. *Di-Me ether*: see under Dimethoxysuccinic Acid. *Dibenzoyl*: needles from EtOH. M.p.  $240^\circ$ .

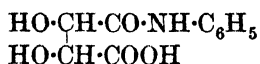
*Di-Me ether*: see under Dimethoxysuccinic Acid.

*Anilide*: see under Tartranilic Acid.

Weerman, *Rec. trav. chim.*, 1917, 37, 45.

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**Tartranilic Acid** (*Tartaric monoanilide*) $\text{C}_{10}\text{H}_{11}\text{O}_5\text{N}$ 

MW, 225

d.

Plates. M.p. 180° decomp. Mod. sol.  $\text{H}_2\text{O}$ , EtOH. (Needles from AcOH. M.p. 194°.)  $[\alpha]_D^{15} + 114.7^\circ$  in MeOH.

$\text{NH}_4$  salt: needles.  $[\alpha]_D^{15} + 102.2^\circ$  in  $\text{H}_2\text{O}$ .

Na salt: needles. M.p. 226°. Spar. sol. EtOH.  $[\alpha]_D^{15} + 101.3^\circ$  in  $\text{H}_2\text{O}$ .

Aniline salt: cryst. M.p. 149–50°.

Me ester:  $\text{C}_{11}\text{H}_{13}\text{O}_5\text{N}$ . MW, 239. Needles from AcOH. M.p. 175°. Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ .  $[\alpha]_D^{15} + 106^\circ$  in MeOH.

Et ester:  $\text{C}_{12}\text{H}_{15}\text{O}_5\text{N}$ . MW, 253. Plates from  $\text{H}_2\text{O}$ . M.p. 151–2°. (Needles, m.p. 163°.) Sol. hot  $\text{H}_2\text{O}$ .  $[\alpha]_D^{15} + 102.4^\circ$  in MeOH.

Propyl ester:  $\text{C}_{13}\text{H}_{17}\text{O}_5\text{N}$ . MW, 267. Plates from EtOH. M.p. 161°.  $[\alpha]_D^{15} + 99.1^\circ$  in MeOH.

Isobutyl ester:  $\text{C}_{14}\text{H}_{19}\text{O}_5\text{N}$ . MW, 281. Needles from EtOH. M.p. 158°.  $[\alpha]_D^{15} + 92.6^\circ$  in MeOH.

Isoamyl ester:  $\text{C}_{15}\text{H}_{21}\text{O}_5\text{N}$ . MW, 295. Needles from EtOH. M.p. 139°.  $[\alpha]_D^{15} + 89.2^\circ$  in MeOH.

Amide:  $\text{C}_{10}\text{H}_{12}\text{O}_4\text{N}_2$ . MW, 224. Anilide of tartramidic acid. Plates. M.p. 226°.  $[\alpha]_D^{15} + 139^\circ$  in  $\text{H}_2\text{O}$ ,  $+ 153^\circ$  in MeOH.

Anilide:  $\text{C}_{16}\text{H}_{16}\text{O}_4\text{N}_2$ . MW, 300. Dianilide of tartaric acid. Prisms from MeOH or needles from EtOH. M.p. 275° (250° decomp., 255–6° decomp., 263–4° decomp.). Sol. Py. Spar. sol.  $\text{Et}_2\text{O}$ , EtOH. Prac. insol.  $\text{C}_6\text{H}_6$ , AcOH. Insol.  $\text{H}_2\text{O}$ .  $[\alpha]_D^{15} + 259^\circ$  in Py ( $[\alpha]_D^{20} + 246.5^\circ$  in Py),  $+ 206^\circ$  in MeOH. Acetyl: needles from EtOH. M.p. 148°. Diacetyl: needles from dil. EtOH. M.p. 214–15°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Prac. insol. ligroin. Insol.  $\text{H}_2\text{O}$ . Tetraacetyl: needles + 2EtOH from EtOH. M.p. 137°.

Di-Me ether: see under Dimethoxysuccinic Acid.

Di-Et ether: see under Diethoxysuccinic Acid.

Tingle, Bates, *J. Am. Chem. Soc.*, 1909, **31**, 1240.

Polikier, *Ber.*, 1891, **24**, 2959.

Bischoff, Nastvogel, *Ber.*, 1890, **23**, 2047.

Arppe, *Ann.*, 1855, **93**, 355.

Casale, *Gazz. chim. ital.*, 1917, **47**, 277.

**Tartronic Acid** (*Hydroxymalonic acid*) $\text{C}_3\text{H}_4\text{O}_5$ 

MW, 120

Prisms +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . Loses  $\text{H}_2\text{O}$  at 60°. M.p. 160° decomp. (156–8°, 155°, 158–9°). Sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Anhyd. acid sol.  $\text{Et}_2\text{O}$ . Sublimes.  $k$  (first) =  $5 \times 10^{-3}$  at 25°. Forms cryst. salts.

Di-Me ester:  $\text{C}_5\text{H}_8\text{O}_5$ . MW, 148. M.p. 44.5–45°. Sol.  $\text{CHCl}_3$ ,  $\text{Et}_2\text{O}$ . Spar. sol. pet. ether.

Di-Et ester:  $\text{C}_7\text{H}_{12}\text{O}_5$ . MW, 176. F.p. –2.5°. B.p. 222–5° (218–19°), 120.5–121°/15 mm.  $\text{D}_4^{15}$  1.152. Et ether: b.p. 228°. Acetyl: b.p. 235–45°, 158–63°/60 mm., 138°/17–18 mm.  $\text{D}_4^{15}$  1.131.

Monoamide:  $\text{C}_3\text{H}_5\text{O}_4\text{N}$ . MW, 119. Needles or prisms from  $\text{H}_2\text{O}$ . M.p. about 160° decomp. Sol. EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ . Prac. insol.  $\text{Et}_2\text{O}$ .

Diamide:  $\text{C}_3\text{H}_6\text{O}_3\text{N}_2$ . MW, 118. Needles from EtOH.Aq. M.p. 195–6° (198°). Mod. sol. hot  $\text{H}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ , EtOH.

Et ether:  $\text{C}_5\text{H}_8\text{O}_5$ . MW, 148. Prisms from pet. ether. M.p. 123–5°. Decomp. at 135°. Sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{C}_6\text{H}_6$ , ligroin.

Wislicenus, Münzesheimer, *Ber.*, 1898, **31**, 552.

Conrad, Brückner, *Ber.*, 1891, **24**, 2997.

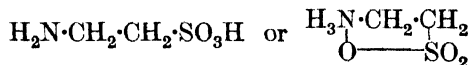
Behrend, Prüsse, *Ann.*, 1918, **416**, 233.

Filippo, *Rec. trav. chim.*, 1910, **29**, 115.

**Tartronylurea.**

See Dialuric Acid.

**Taurine** (*Aminoethylsulphonic acid, aminoethanesulphonic acid, ethylaminesulphonic acid*)

 $\text{C}_2\text{H}_7\text{O}_3\text{NS}$ 

MW, 125

Occurs mainly combined. Free in lungs and flesh extract of oxen. In shark blood; liver, spleen and kidney of ray; in muscle, oysters, molluscs, etc. Columns from  $\text{H}_2\text{O}$ . Decomp. at 300–5°. Sol. 15.5 parts  $\text{H}_2\text{O}$  at 12°. Insol. EtOH. Neutral in dil. sol. Acid in conc. sol. Heat of comb.  $\text{C}_p$  382.2 Cal.,  $\text{C}_v$  382.9 Cal. Stable to boiling conc. acids. Not esterified readily. Phenol + hypochlorites  $\rightarrow$  blue col.  $\text{MeOH} + \text{KOH} + \text{MeI} \rightarrow$  taurobetaine. Hg salt used for identification and separation.

N-Me:  $\text{C}_3\text{H}_9\text{O}_3\text{NS}$ . MW, 139. Prisms. M.p. 241–2°. Very sol.  $\text{H}_2\text{O}$ . Insol. EtOH,  $\text{Et}_2\text{O}$ .

N-Di-Me:  $\text{C}_4\text{H}_{11}\text{O}_3\text{NS}$ . MW, 153. Prisms from MeOH. M.p. 315–16°. Very sol.  $\text{H}_2\text{O}$ , AcOH. Insol. EtOH,  $\text{Et}_2\text{O}$ .

N-Et:  $\text{C}_4\text{H}_{11}\text{O}_3\text{NS}$ . MW, 153. Prisms from  $\text{H}_2\text{O}$ . M.p. 147°.

*N-Di-Et*:  $C_6H_{15}O_3NS$ . MW, 181. Plates from EtOH. M.p.  $151^\circ$ .

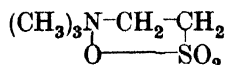
*N-Allyl*:  $C_5H_{11}O_3NS$ . MW, 165. Prisms from EtOH. M.p.  $190-5^\circ$ .

Cortese, *J. Am. Chem. Soc.*, 1936, **58**, 191.

Teroaka, *Z. physiol. Chem.*, 1925, **145**, 238.

James, *J. prakt. Chem.*, 1885, **31**, 414.

#### Taurobetaine (Trimethyltaurine)



$C_5H_{13}O_3NS$

MW, 167

Prisms from  $H_2O$ . Does not melt below  $250^\circ$ . Very sol.  $H_2O$ . Insol. EtOH,  $Et_2O$ . Neutral reaction. Sweet taste.  $Ba(OH)_2 \rightarrow$  trimethylamine + isethionic acid.

Brieger, *Z. physiol. Chem.*, 1882, **7**, 36.

**Taurocarbamic Acid** (2-Ureidoethane-1-sulphonic acid, sulphoethylurea)



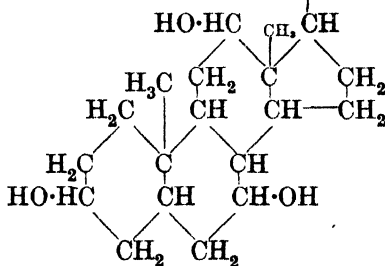
$C_3H_8O_4N_2S$

MW, 168

Prisms from EtOH.Aq. M.p.  $182^\circ$  decomp. Very sol.  $H_2O$ . Spar. sol. EtOH. Insol.  $Et_2O$ .  $Ba(OH)_2$  or HCl on heating  $\rightarrow$  taurine. Forms cryst. Ba and Ag salts.

Lippich, *Z. physiol. Chem.*, 1910, **68**, 292.

#### Taurocholic Acid



$C_{26}H_{45}O_7NS$

MW, 515

Constituent of bile. Amorph. powder. M.p. about  $125^\circ$  decomp. Very sol.  $H_2O$ , hot EtOH. Spar. sol.  $Et_2O$ , AcOEt. Very hygroscopic.  $[\alpha]_D^{20} + 38.8^\circ$  in EtOH.Aq.

*Na salt*: needles or plates +  $1\frac{1}{2}H_2O$  from  $H_2O$ . M.p. anhyd.  $180^\circ$ . Boiling  $H_2O \rightarrow$  isomeric form, cryst. +  $2H_2O$ , m.p.  $235^\circ$ . Both forms have identical solubilities and rotations.

*Ba salt*: plates or needles +  $5H_2O$  from  $H_2O$ .

M.p.  $225-7^\circ$  decomp. Very sol.  $H_2O$ , hot EtOH. Spar. sol.  $Et_2O$ , AcOEt.  $[\alpha]_D^{20} + 25.6^\circ$ .

Kazuno, Yamasaki, *Z. physiol. Chem.*, 1934, **224**, 160.

Tanaka, *Z. physiol. Chem.*, 1933, **220**, 39.

Hammarsten, *Z. physiol. Chem.*, 1904, **43**, 127.

#### Tazettine (Ungernine)

$C_{18}H_{21}O_5N$

MW, 331

Constituent of bulbs of *Narcissus tazetta*. After high vac. sublimation, m.p.  $210-11^\circ$ . Sol. EtOH. Spar. sol.  $Et_2O$ .  $[\alpha]_D^{15} + 150.4$  in  $CHCl_3$ .  $H_2SO_4 \rightarrow$  brownish-red col. Zn dust dist.  $\rightarrow$  phenanthridine.  $KMnO_4 \rightarrow$  hydrastatic acid.

*Acetyl deriv.*: cryst. from  $Et_2O$ -pet. ether. M.p.  $125-126.5^\circ$ .

*Methiodide*: m.p.  $220^\circ$  decomp.

*Picrate*: yellow cryst. from EtOH. M.p.  $205-8^\circ$  decomp.

*Perchlorate*: m.p.  $105-8^\circ$  decomp.  $[\alpha]_D^{15} + 109.6^\circ$  in MeOH.

Robinson, *Ann. Rev. Biochem.*, 1935, **4**, 507.

Späth, Kahovec, *Ber.*, 1934, **67**, 1501.

Norkina, Orechhoff, *Ber.*, 1936, **69**, 500.

Späth, Orechhoff, Kuffner, *ibid.*, 2446.

#### Tecomin.

See Lapachol.

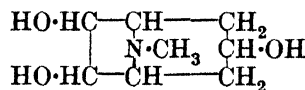
#### Tectochoyrsin.

See under Chrysin.

#### Tectoquinone.

See under 2-Methylantraquinone.

**Teloidine** (3 : 6 : 7-Trihydroxytropane, tropantriol-3 : 6 : 7)



$C_8H_{15}O_3N$

MW, 173

Needles +  $1H_2O$  from  $Me_2CO$ .Aq. M.p.  $168-9^\circ$ . Very sol.  $H_2O$ , EtOH. Spar. sol. most cold org. solvents.

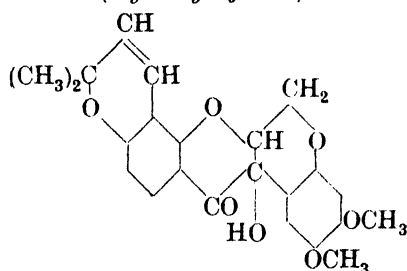
*B.HCl*: prisms from EtOH. Does not melt below  $300^\circ$ .

*B.HBr*: plates and needles from EtOH. M.p.  $295^\circ$  decomp.

*B.HAuCl<sub>4</sub>*: yellow plates +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p.  $225^\circ$  decomp.

King, *J. Chem. Soc.*, 1919, **115**, 487.

Pyman, Reynolds, *J. Chem. Soc.*, 1908, **93**, 2079.

**Tephrosin (Hydroxydeguelin)** $C_{23}H_{22}O_7$ 

MW, 410

Constituent of derris root, cubé root (*Lonchocarpus nicou*) and leaves of *Cracca vogeli*. Prisms from MeOH-CHCl<sub>3</sub>. M.p. 198°. Ox. → tephrosin-dicarboxylic acid.

Acetyl: cryst. from MeOH.Aq. M.p. 200°.

Clark, *J. Am. Chem. Soc.*, 1933, **55**, 759; 1932, **54**, 3000; 1931, **53**, 729.

Takei, Mayujima, Ono, *Chem. Abstracts*, 1933, **27**, 2954.

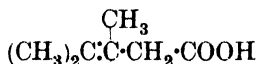
Clark, Cloborn, *J. Am. Chem. Soc.*, 1932, **54**, 4454.

Butenandt, Hilgetag, *Ann.*, 1932, **495**, 172.

**Teraconic Acid.**

See Isopropylidenesuccinic Acid.

**Teracrylic Acid** (2 : 3-Dimethyl-2-pentene-5-carboxylic acid, 2-isopropylidene-butyrac acid)

 $C_7H_{12}O_2$ 

MW, 128

Liq. B.p. 218°. Spar. sol. H<sub>2</sub>O.

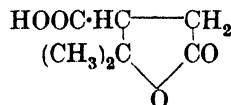
Et ester:  $C_9H_{16}O_2$ . MW, 156. Liq. with fruity odour. B.p. 189-91°.

Fittig, Kraft, *Ann.*, 1881, **208**, 79.

**Tereanilic Acid.**

See 2 : 5-Diaminoterephthalic Acid.

**Terebic Acid** (2 : 2-Dimethylparaconic acid, 3 : 3-dimethylbutyrolactone-2-carboxylic acid)

 $C_7H_{10}O_4$ 

MW, 158

Cryst. from EtOH. M.p. 175°. Sol. boiling H<sub>2</sub>O, warm EtOH. Spar. sol. cold H<sub>2</sub>O. Heat of comb.  $C_v$  778.4 Cal.  $k = 2.65 \times 10^{-4}$  at 25°.

Me ester:  $C_8H_{12}O_4$ . MW, 172. B.p. 148-9°/17 mm.

Et ester:  $C_9H_{14}O_4$ . MW, 186. B.p. 273-5°, 145-7°/15 mm.

Chloride:  $C_7H_9O_3Cl$ . MW, 176.5. B.p. 143°/12 mm.

Anilide: cryst. from EtOH. M.p. 176°.

Barbier, Locquin, *Bull. soc. chim.*, 1913, **13**, 231.

Simonsen, *J. Chem. Soc.*, 1907, **91**, 186.

**Terephthalaldehyde** (Terephthalaldehyde, 1 : 4-dialdehydobenzene)

 $C_8H_6O_2$ 

MW, 134

Needles from H<sub>2</sub>O. M.p. 116°. B.p. 245-8°/771 mm. Sol. 5000 parts cold H<sub>2</sub>O, 80 parts hot H<sub>2</sub>O. Sol. Et<sub>2</sub>O. Very sol. EtOH. Sol. alkalis, pptd. by acids. Sublimes. Difficultly volatile in steam.  $K_2Cr_2O_7 + H_2SO_4 \rightarrow p$ -aldehydobenzoic acid.  $Ac_2O + H_2SO_4 \rightarrow$  tetraacetate.

Dioxime: terephthalaldoxime.  $C_8H_8O_2N_2$ . MW, 164. Cryst. M.p. 200°. Spar. sol. H<sub>2</sub>O. Very sol. EtOH, Et<sub>2</sub>O. Di-Et ether: cryst. M.p. 55°. Diacetyl: cryst. M.p. 155°.

Dihydrazone: yellowish-white cryst. from EtOH. M.p. 165° decomp.

Monophenylhydrazone: orange-yellow needles from Et<sub>2</sub>O. M.p. 152-4°.

Di-phenylhydrazone: yellow scales from AcOH or acetoacetic ester. M.p. 278° decomp.

Di-p-nitrophenylhydrazone: cryst. from PhNO<sub>2</sub>. Sinters at 272°, m.p. 281°.

Adams, Bullock, Wilson, *J. Am. Chem. Soc.*, 1923, **45**, 521.

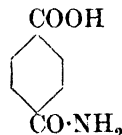
Rosenmund, Zetzsche, *Ber.*, 1921, **54**, 2888.

Thiele, Günther, *Ann.*, 1906, **347**, 110.

**Terephthalaldehydic Acid.**

See p-Aldehydobenzoic Acid.

**Terephthalamic Acid** (Terephthalic acid monoamide)

 $C_8H_7O_3N$ 

MW, 165

Does not melt below 300°. Begins to sublime at 250°. Insol. hot H<sub>2</sub>O and most org. solvents. Hyd. → terephthalic acid.

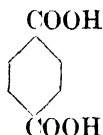
Me ester:  $C_9H_9O_3N$ . MW, 179. Cryst. from hot H<sub>2</sub>O. M.p. 201°.



**Nitrile**:  $C_8H_6ON_2$ . MW, 146. Needles. M.p. 223°.

Kattwinkel, Wolfenstein, *Ber.*, 1904, 37, 3222.

**Terephthalic Acid** (*p*-Phthalic acid, benzene-1:4-dicarboxylic acid)



$C_8H_6O_4$

MW, 166

Needles. Sublimes without melting at about 300°. Sol. 67,000 parts cold  $H_2O$ . Insol.  $Et_2O$ ,  $EtOH$ ,  $AcOH$ ,  $CHCl_3$ . Sol. hot  $EtOH$ . Heat of comb.  $C_p$  770.9 Cal.,  $C_r$  771.2 Cal. Gives spar. sol. Ca, Sr and Ba salts. Red.  $\rightarrow$  *cis*- and *trans*-hexahydroterephthalic acids.

**Mono-Me ester**:  $C_9H_8O_4$ . MW, 180. Needles from hot  $H_2O$ . M.p. about 230°. Sublimes.

**Di-Me ester**:  $C_{10}H_{10}O_4$ . MW, 194. Needles from  $Et_2O$ . M.p. 141–2°. Sol. 300 parts hot  $H_2O$ . Spar. sol. cold  $EtOH$ . Sublimes. Volatile in steam.

**Mono-Et ester**:  $C_{10}H_{10}O_4$ . MW, 194. Plates from  $C_6H_6$ . M.p. 171°.

**Di-Et ester**:  $C_{12}H_{14}O_4$ . MW, 222. Prisms from  $EtOH$  or pet. ether. M.p. 44°. B.p. 302°, 142°/2 mm.  $D_4^{25}$  1.1098. Sol.  $Et_2O$ . Very sol. cold  $EtOH$ . Insol.  $H_2O$ .

**Mono-propyl ester**:  $C_{11}H_{12}O_4$ . MW, 208. M.p. 127–9°.

**Dipropyl ester**:  $C_{14}H_{18}O_4$ . MW, 250. Needles or plates from  $C_6H_6$ . M.p. 31°. B.p. 158°/4 mm. Very sol. hot  $EtOH$ ,  $Et_2O$ .

**Mono-isopropyl ester**: needles from hot  $C_6H_6$ . M.p. 166°. Very sol.  $EtOH$ ,  $Et_2O$ . Insol.  $H_2O$ .

**Di-isopropyl ester**: plates. M.p. 55–6°.

**Mono-butyl ester**:  $C_{12}H_{14}O_4$ . MW, 222. M.p. 122–4°.

**Dibutyl ester**:  $C_{18}H_{22}O_4$ . MW, 278. Needles. M.p. 16°. B.p. 180°/4 mm.

**Mono-isobutyl ester**: m.p. 151–4°.

**Di-isobutyl ester**: plates. M.p. 55°. B.p. 180°/6 mm.

**Di-tert.-butyl ester**: prisms from  $MeOH$ . M.p. 118°.

**Di-1-menthyl ester**:  $C_{28}H_{42}O_4$ . MW, 442. Needles from  $EtOH$ . M.p. 77–8°.  $[\alpha]_D^{20}$  –102.6° in  $CHCl_3$ .

**Diphenyl ester**:  $C_{20}H_{14}O_4$ . MW, 318. Needles from  $EtOH$ . M.p. 191°.

**Monochloride**:  $C_8H_5O_3Cl$ . MW, 184.5. Needles from  $C_6H_6$ . Does not melt below 300°.

**Dichloride**:  $C_8H_4O_2Cl_2$ . MW, 203. Needles or plates from ligroin. M.p. 83–4°.

**Dibromide**:  $C_8H_4O_2Br_2$ . MW, 292. Needles from pet. ether. M.p. 85°.

**Mono-amide**: see Terephthalamide.

**Diamide**:  $C_8H_8O_2N_2$ . MW, 164. Plates from  $AcOH$ . Needles from  $H_2O$ . Does not melt below 250°.

**Di-1-naphthylamide**: needles from  $PhNO_2$ . Sinters at 325°, m.p. 334–5°.

**Di-diphenylamide**: needles from xylene. Sinters at 268°, m.p. 272–3° decomp.

**Di-azide**: plates from  $Me_2CO$ . M.p. 110°.

**Di-hydrazide**: needles from  $H_2O$ . Does not melt below 300°.

**Mono-nitrile**: see *p*-Cyanobenzoic Acid.

**Di-nitrile**: 1:4-dicyanobenzene.  $C_8H_4N_2$ . MW, 128. Needles from  $H_2O$  or  $MeOH$ . M.p. 222°. Very sol. hot  $AcOH$ . Spar. sol.  $EtOH$ ,  $Et_2O$ . Insol. cold  $H_2O$ . Sublimes.

**Dianilide**: needles from acetoacetic ester or  $PhNO_2$ . M.p. 334–7°.

Carré, Libermann, *Compt. rend.*, 1934, 199, 1422.

Smith, *J. Am. Chem. Soc.*, 1921, 43, 1920. Rosenmund, Zetzsche, *Ber.*, 1921, 54, 2888.

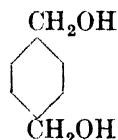
Lyons, Reid, *J. Am. Chem. Soc.*, 1917, 39, 1740.

Cohen, Pennington, *J. Chem. Soc.*, 1918, 113, 63.

### Terephthalophenone.

See *p*-Dibenzoylbenzene.

**Terephthalyl Alcohol** (*p*-Xylylene glycol,  $\omega$ -dihydroxy-*p*-xylene)



$C_8H_{10}O_2$

MW, 138

Needles. M.p. 115–16°. Sol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ .

**Mono-Me ether**:  $C_9H_{12}O_2$ . MW, 152. B.p. 152°/16 mm.  $D_4^{17}$  1.076.  $n_D^{17}$  1.529. **Acetyl**: b.p. 150°/16 mm.  $D_4^{20}$  1.080.  $n_D^{20}$  1.505. **Phenylurethane**: m.p. 62°.

**Di-Me ether**:  $C_{10}H_{14}O_2$ . MW, 166. Liq. B.p. 235°, 124°/18 mm.  $D_4^{18}$  1.013.  $n_D^{18}$  1.503.

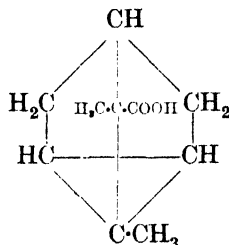
**Mono-Et ether**:  $C_{10}H_{14}O_2$ . MW, 166. Liq. B.p. 250–2°, 154°/16 mm.  $D_4^{17}$  1.047.  $n_D^{17}$  1.520. Sol.  $EtOH$ ,  $Et_2O$ . Insol.  $H_2O$ .

**Di-Et ether**:  $C_{12}H_{18}O_2$ . MW, 194. Liq. B.p. 251–2°/734 mm., 134°/15 mm.  $D_4^{18}$  0.976.  $n_D^{18}$  1.493.

*Dibenzyl ether*:  $C_{22}H_{22}O_2$ . MW, 318. M.p. 67°.

Quelet, *Bull. soc. chim.*, 1933, **53**, 222.

### Teresantalic Acid



$C_{10}H_{14}O_2$

MW, 166

Occurs in sandalwood oil. Prisms from EtOH. M.p. 158°. B.p. 157–8°/20 mm. Sol. 1270 parts  $H_2O$  at 20°.  $[\alpha]_D^{20} = -76.6^\circ$  in  $C_6H_6$ . Volatile in steam. Stable to  $KMnO_4$ . Forms a hydrochloride. Dil.  $H_2SO_4 \rightarrow$  santene.

*Ca salt*: cryst. +  $1\frac{1}{2}H_2O$  from  $H_2O$ . Decomp. at 100°.

*Hydrochloride*: cryst. from MeOH. M.p. 199°. Spar. sol. pet. ether.

*Me ester*:  $C_{11}H_{16}O_2$ . MW, 180. Oil. B.p. 88.5–89.5°/13 mm.  $D_4^{20} 1.0305$ .  $[\alpha]_D^{20} = -60.79^\circ$ .  $Na + EtOH \rightarrow$  teresantalol. *Hydrochloride*: two forms. (i) Leaflets from EtOH.Aq. M.p. 68°. B.p. 125–7°/10 mm.  $[\alpha]_D^{20} = +9.22^\circ$  in  $C_6H_6$ . (ii) Oil. *Hydrobromide*: oil. F.p.  $-10^\circ$ . M.p. 20°.

*Chloride*:  $C_{10}H_{13}OCl$ . MW, 184.5. B.p. 100–4°/14 mm.

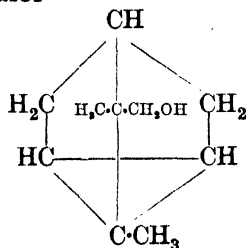
*Anilide*: b.p. 204–5°/11 mm.

Asahina, Ishidate, Momose, *Ber.*, 1935, **68**, 83.

Ruzicka, Liebl, *Helv. Chim. Acta*, 1926, **9**, 140.

Rupe, Tomi, *Ber.*, 1916, **49**, 2556.

### Teresantalol



$C_{10}H_{16}O$

MW, 152

*d.*

Constituent of Indian sandalwood oil. Prisms from pet. ether. M.p. 112–14°. B.p. 95–8°/9 mm.  $[\alpha]_D + 11.58^\circ$  in EtOH. Sublimes.

*Acetyl*: b.p. 102–3°/9–10 mm.  $D_4^{20} 1.019$ .  $n_D^{20} 1.470$ .  $[\alpha]_D + 21^\circ$ .

*dl.*

Prisms from  $C_6H_6$ . M.p. 118°. B.p. 97–8°/10 mm. Stable to cold  $KMnO_4$ .

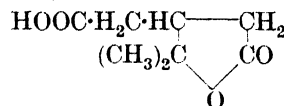
Asahina, Ishidate, *Ber.*, 1935, **68**, 952.

Semmler, Bartelt, *Ber.*, 1907, **40**, 3103.

### Terpane.

See *p*-Menthane.

**Terpenylic Acid** (3 : 3-Dimethylbutyrolactone-2-acetic acid)



$C_8H_{12}O_4$

MW, 172

Plates or prisms +  $1H_2O$  from  $H_2O$ . M.p. 57°, anhyd. 90°. Very sol. hot  $H_2O$ . Mod. sol. cold  $H_2O$ . Sublimes at 130–40°. Ox.  $\rightarrow$  terebic acid.  $HI (+ P) \rightarrow$  2-isopropylglutaric acid.

*Me ester*:  $C_9H_{14}O_4$ . MW, 186. B.p. 145–7°/15 mm.

*Et ester*:  $C_{10}H_{16}O_4$ . MW, 200. M.p. 37–5°. B.p. 305°, 174–7°/15 mm.

Lawrence, *J. Chem. Soc.*, 1899, **75**, 530.

Wallach, *Ann.*, 1893, **277**, 118.

Fittig, Levy, *Ann.*, 1890, **256**, 109.

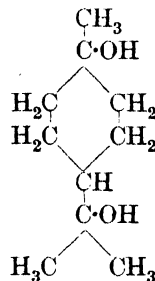
### *m*-Terphenyl.

1 : 3-Diphenylbenzene, *q.v.*

### *p*-Terphenyl.

1 : 4-Diphenylbenzene, *q.v.*

### Terpin (*p*-Menthandiol-1 : 8)



$C_{10}H_{20}O_2$

MW, 172

*Cis*:

Cryst. M.p. 105.5°. B.p. 258°. Absorbs moisture from the air. Heat of comb.  $C_v$  1454.4 Cal. Dist. over  $Al_2O_3$  at 200–80°  $\rightarrow$  dipentene.  $H (+ Ni) \rightarrow$  *p*-cymene.  $(COOH)_2 \rightarrow$  *d*-terpineol.

*Diformyl*: viscous oil. B.p. 176–7°/40 mm.  $D_4^{27} 1.067$ .

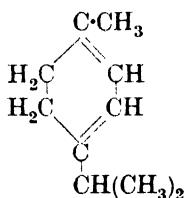
*Diacetyl*: b.p. 145°/14 mm.

*Trans* :

Prisms or plates from AcOEt. M.p. 156–8°. B.p. 263–5°. Very sol. EtOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, AcOEt.

Aschan, *Chem. Zentr.*, 1919, I, 284.

Wallach, *Ann.*, 1906, **350**, 154.

 $\alpha$ -Terpinene ( $\Delta^{1,3}$ -*p*-Menthadiene)C<sub>10</sub>H<sub>16</sub>

MW, 136

Constituent of many essential oils. Oil with lemon odour. B.p. 173.5–174.8°/755 mm. D<sub>4</sub><sup>20</sup> 0.8375. n<sub>D</sub><sup>20</sup> 1.477. Resinifies on keeping. Heat of comb. C<sub>10</sub> 1469.9 Cal.

*Di-hydrochloride* : terpinene dihydrochloride. Cryst. from MeOH. M.p. 53–4°. B.p. 108–9°/10 mm.

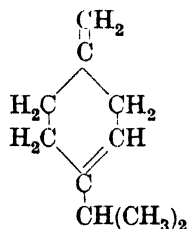
*Di-hydrobromide* : terpinene dihydrobromide. Plates from MeOH or AcOH. M.p. 58–9°.

*Di-hydriodide* : terpinene dihydriodide. Cryst. from MeOH. M.p. 76°.

*Nitrosite* : prisms. M.p. 155°. *Benzoate* : m.p. 77–8°.

Richter, Wolff, *Ber.*, 1930, **63**, 1714.

Wallach, *Ann.*, 1889, **252**, 133; 1887, **241**, 316; 1887, **239**, 35.

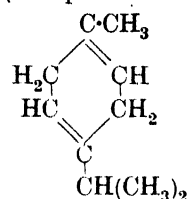
 $\beta$ -Terpinene ( $\Delta^{3,1(7)}$ -*p*-Menthadiene)C<sub>10</sub>H<sub>16</sub>

MW, 136

Does not occur naturally. Oil. B.p. 173–4°. D<sub>20</sub><sup>20</sup> 0.838. n<sub>D</sub><sup>20</sup> 1.4754. Readily oxidised in air. HCl → terpinene dihydrochloride.

*Tetrabromide* : prisms from AcOEt. M.p. 154–5°.

Wallach, *Ann.*, 1908, **362**, 288.

 $\gamma$ -Terpinene ( $\Delta^{1,4}$ -*p*-Menthadiene)C<sub>10</sub>H<sub>16</sub>

MW, 136

Constituent of a few essential oils. B.p. 183°, 72.5°/18 mm. D<sub>4</sub><sup>20</sup> 0.849. n<sub>D</sub><sup>14.5</sup> 1.4765. Heat of comb. C<sub>10</sub> 1468.9 Cal., C<sub>p</sub> 1472 Cal. HCl → terpinene dihydrochloride.

*Tetrabromide* : plates from pet. ether. M.p. 128°.

*Nitroschloride* : m.p. 111°.

*Nitrolpiperidide* : plates. M.p. 149°.

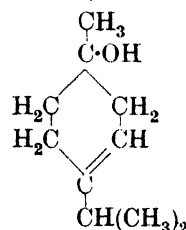
*Nitrosate* : needles from AcOH–MeOH. M.p. 116° decomp.

Richter, Wolff, *Ber.*, 1930, **63**, 1714.

**Terpinene-terpin.**

See *p*-Menthandiol-1 : 4.

(1)-Terpinenol ( $\Delta^3$ -*p*-Menthenol-1, 4-methyl-1-isopropylcyclohexenol-4)

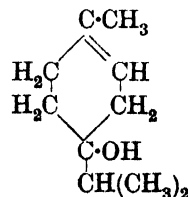
C<sub>10</sub>H<sub>18</sub>O

MW, 154

B.p. 208–10°. D<sub>18</sub><sup>18</sup> 0.9265. n<sub>D</sub><sup>18</sup> 1.4781. KMnO<sub>4</sub> → *p*-menthantriol-1 : 3 : 4.

Wallach, Meister, *Ann.*, 1908, **362**, 269.

(4)-Terpinenol ( $\Delta^1$ -*p*-Menthenol-4, 1-methyl-4-isopropylcyclohexenol-4)

C<sub>10</sub>H<sub>18</sub>O

MW, 154

d-.

Occurs in many essential oils. B.p. 208–10°, 93–6°/1 mm. D<sub>20</sub><sup>20</sup> 0.926. n<sub>D</sub><sup>19</sup> 1.4785. [α]<sub>D</sub><sup>11</sup> +24.5°. Dil. H<sub>2</sub>SO<sub>4</sub> → *p*-menthandiol-1 : 4. KMnO<sub>4</sub> → *d*-menthantriol-1 : 2 : 4.

*Formyl*: b.p. 102–6°.  $D_4^{20}$  0.975.  $n_D$  1.4745.

*Nitrosochloride*: m.p. 111–12°.

*Nitrolpiperidide*: m.p. 172–4°.

*Phenylurethane*: m.p. 71–2°.

1-Naphthylurethane: m.p. 105.5–106.5°.

*dl.*

Constituent of oil from leaf of *Liquidambar formosana*, Hance. B.p. 212–14°, 90°/11 mm.  $n_D$  1.4803.

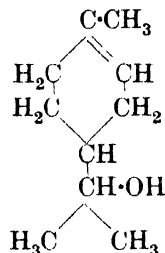
*Nitrolpiperidide*: Two forms. (i) Cryst. from EtOH. M.p. 155–6°. (ii) Cryst. from EtOH. M.p. 181–2°. More soluble.

Kafuku, Nonoe, Hata, *J. Chem. Soc. Japan*, 1934, 55, 224.

Wallach, Meister, *Ann.*, 1908, 362, 269.

Wallach, *Ber.*, 1907, 40, 596.

#### $\alpha$ -Terpineol ( $\Delta^1$ -p-Menthenol-8)



$C_{10}H_{18}O$

MW, 154

All forms occur in many essential oils.

*d.*

M.p. 36.9°. B.p. 104°/15 mm.  $D_4^{21.5}$  0.9475.  $n_D$  1.4819.  $[\alpha]_D^{20} + 100.5^\circ$ .

*Nitrosochloride*: leaflets from MeOH. M.p. 107–8°.

*Nitrolpiperidide*: m.p. 151–2°.

*Formate*: b.p. 133–6°/40 mm.  $D_0$  0.9989.  $[\alpha]_D + 16.5^\circ$ .

*Acetate*: b.p. 140°/40 mm.  $D_0$  0.9828.  $[\alpha]_D + 52.5^\circ$ .

*Hydrogen phthalate*:  $[\alpha]_D + 36.7^\circ$ . *Morphine salt*: rhombic cryst. from MeOH. M.p. 142°.

*Phenylurethane*: needles from MeOH. M.p. 109.5°.

*l.*

M.p. 37.0°. B.p. 104°/15 mm.  $D_4^{25}$  0.9364.  $n_D^{25}$  1.48054.  $[\alpha]_D^{20} - 100.5^\circ$ .

*Nitrosochloride*: needles from MeOH.Aq. M.p. 107–8°.

*Nitrolpiperidide*: m.p. 151–2°.

*Formate*: b.p. 135–8°/40 mm.  $D_0$  0.9986.  $[\alpha]_D - 69.4^\circ$ .

*Hydrogen phthalate*:  $[\alpha]_D - 36.7^\circ$ . *Strychnine salt*: needles from EtOH. M.p. 207°.

*Phenylurethane*: m.p. 109.5°.

*dl.*

M.p. 35°. B.p. 218.8–219.4°/752 mm., 99–100°/12 mm.  $D_4^{20}$  0.935.  $n_D^{20}$  1.4819. Non misc. with  $H_2O$ . Misc. with most org. solvents. Heat of comb.  $C_p$  1475.1 Cal.  $KMnO_4 \rightarrow p$ -menthantriol-1 : 2 : 8 and terpenylic acid.  $H_2O$  at 250°  $\rightarrow$  dipentene.  $H\cdot COOH \rightarrow$  terpinolene.  $H(+Ni) \rightarrow p$ -cymene,  $H(+Pd) \rightarrow p$ -menthanol-8.

*Nitrosochloride*: needles from AcOEt. Woolly needles from MeOH. M.p. 120–2°.

*Nitrolpiperidide*: m.p. 159–60°.

*Me ether*:  $C_{11}H_{20}O$ . MW, 168. B.p. 212°.

*Acetate*: b.p. 104–6°/11 mm.  $D_4^{20}$  0.9659.  $n_D^{19.7}$  1.4657.

*Hydrogen phthalate*: rods from AcOH. M.p. 117–18°. *Brucine salt*: rhombic cryst. from MeOH. M.p. 150°.

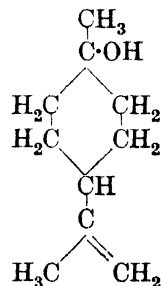
*Phenylurethane*: m.p. 113°.

Ransac, *Chem. Zentr.*, 1932, I, 63.

Fuller, Kenyon, *J. Chem. Soc.*, 1924, 125, 2304.

Perkin, *J. Chem. Soc.*, 1904, 85, 665.

#### $\beta$ -Terpineol ( $\Delta^{8(9)}$ -p-Menthenol-1)



$C_{10}H_{18}O$

MW, 154

Does not occur naturally. Oil with odour of hyacinths. Solidifies on cooling to needles, m.p. 32–3°. B.p. 209–10°/752 mm., 90°/10 mm.  $D_4^{20}$  0.919.  $n_D^{20}$  1.4747.  $KMnO_4 \rightarrow p$ -menthantriol-1 : 8 : 9.

*Dibromide*: needles from EtOH–Et<sub>2</sub>O. M.p. 114–15°.

*Nitrosite*: needles from Me<sub>2</sub>CO.Aq. M.p. 78°.

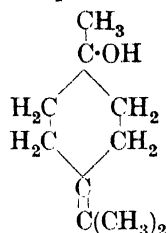
*Nitrosate*: needles from EtOH.Aq. M.p. 125°.

*Nitrosochloride*: cryst. from EtOH.Aq. M.p. 103°.

*Nitrolanilide*: m.p. 145–6°.

*Phenylurethane*: m.p. 85°.

See first reference above and also Stephen, Helle, *Ber.*, 1902, 35, 2149.

$\gamma$ -Terpineol ( $\Delta^{4(8)}$ -*p*-Menthenol-1) $C_{10}H_{18}O$ 

MW, 154

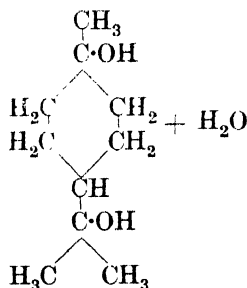
Thick prisms from  $Et_2O$ . M.p. 68–70°. Distills without decomp.  $KMnO_4 \rightarrow p$ -menthatriol-1 : 4 : 8.  $H-COOH \rightarrow$  terpinolene.

*Dibromide*: m.p. 114–15°.

*Acetyl*: b.p. 110–20°/16 mm. *Dibromide*: m.p. 103°. *Nitroschloride*: blue leaflets from  $EtOH$ . M.p. 82°. *Nitrosobromide*: blue needles from  $EtOH$ . M.p. 81–2°.

Baeyer, *Ber.*, 1894, **27**, 443.

## Terpin Hydrate

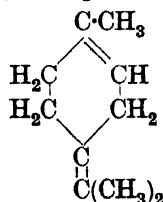
 $C_{10}H_{20}O_2 \cdot H_2O$ 

MW, 190

Rhombic cryst. M.p. 123° decomp.  $\rightarrow$  *cis*-terpin. Eutectic containing 10% hydrate has m.p. 95°. Sol. 250 parts  $H_2O$  at 15°, 32 parts at 100°. Sol. 10 parts  $EtOH$  at 15°, 2 parts at 78°. Sol. 100 parts  $Et_2O$ , 200 parts  $CHCl_3$  at 15°. Sol. 1 part of  $AcOH$  at b.p. Insol. pet. ether.  $H_2SO_4 \rightarrow$  orange col. Heat of comb.  $C_v$  1449.1 Cal.

Claus, *Chem. Abstracts*, 1932, **26**, 127.

Aschan, *Chem. Zentr.*, 1919, **I**, 284.

Terpinolene ( $\Delta^{1,4(8)}$ -*p*-Menthadiene) $C_{10}H_{16}$ 

MW, 136

Naturally occurring terpene hydrocarbon. B.p. 186°, 121°/111 mm., 76°/10 mm. Poly-

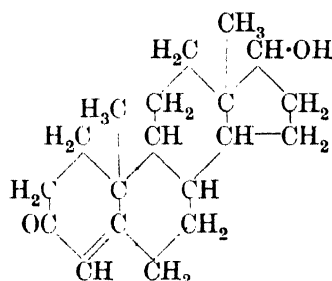
merises when dist. at atm. press. Very readily isomerises to terpinene.  $D_{15}^{20} 0.8633$ .  $n_D^{20} 1.4883$ .  $HCl \rightarrow$  dipentene dihydrochloride.  $Ac_2O + H_2SO_4 \rightarrow$  transient pink col.

*Dibromide*: prisms. M.p. 69–70°.

*Tetrabromide*: two forms. They show marked m.p. depression but give same terpinolene on debromination. (i) Rectangular plates from  $Me_2CO$ . M.p. 119°. (ii) Cryst. aggregates from  $EtOH$ ,  $Me_2CO$  or  $AcOEt$ . M.p. 122°.

Henry, Paget, *J. Chem. Soc.*, 1931, **134**, 28.

Wallach, *Ann.*, 1909, **368**, 11.

Testosterone (*Androstenol-17-one-3*) $C_{19}H_{28}O_2$ 

MW, 288

Hormone obtained from testes extract. Needles from  $Me_2CO$ . M.p. 154–154.5°.  $[\alpha]_D^{25} + 109^\circ$  in  $EtOH$ . Produces male characteristics in castrated animals.

*Oxime*: cryst. from  $EtOH.Aq$ . M.p. 222–3°.

*Formyl*: cryst. from hexane. M.p. 127–9°.

*Acetyl*: needles from  $Me_2CO.Aq$ . M.p. 140–1°.  $[\alpha]_D^{25} + 87.8^\circ$  in  $EtOH$ .

*Propionyl*: m.p. 121–3°.

*Butyryl*: needles from 70%  $MeOH$ . M.p. 111–13°.

*Isobutyryl*: needles from  $Me_2CO.Aq$ . M.p. 134–6°.

*Palmityl*: cryst. from  $MeOH$ . M.p. 72–4°.

*Stearyl*: cryst. from  $MeOH$ . M.p. 79–80°.

David, Dingemance, Freud, Laquer, *Z. physiol. Chem.*, 1935, **233**, 281.

Butenandt, Hanisch, *Ber.*, 1935, **68**, 1859.

Ruzicka, Wettstein, *Helv. Chim. Acta*, 1935, **18**, 1264; 1936, **19**, 1141.

## Tetanthrene.

See Tetrahydrophenanthrene.

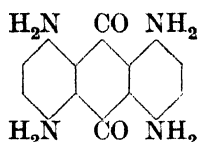
## Tethracene.

See Tetrahydroanthracene.

## Tethracenequinone.

See 1 : 2 : 3 : 4-Tetrahydroanthraquinone.

## 1 : 4 : 5 : 8-Tetra-aminoanthraquinone

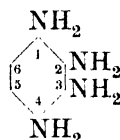
C<sub>14</sub>H<sub>12</sub>O<sub>2</sub>N<sub>4</sub> MW, 268

Reddish-brown needles from EtOH. M.p. 332°. Sol. EtOH and CHCl<sub>3</sub> with greenish-blue col., in AcOEt with reddish-blue col., in Me<sub>2</sub>CO with blue col. Spar. sol. H<sub>2</sub>O with blue col.

1 : 4 : 5 : 8-N-Tetra-Me : C<sub>18</sub>H<sub>20</sub>O<sub>2</sub>N<sub>4</sub>. MW, 324. Cryst. Sol. CHCl<sub>3</sub>, AcOEt with blue col. Sol. HCl with red col. turning violet on dilution.

Noelting, Wortmann, *Ber.*, 1906, **39**, 644.  
Höchst, D.R.P., 156,803, (*Chem. Zentr.*, 1905, I, 313).

## 1 : 2 : 3 : 4-Tetra-aminobenzene

C<sub>6</sub>H<sub>10</sub>N<sub>4</sub> MW, 138

Free base oxidises very rapidly in air.

Nietzki, Geese, *Ber.*, 1899, **32**, 505.

## 1 : 2 : 3 : 5-Tetra-aminobenzene.

B,3HCl : needles + H<sub>2</sub>O.

B,2H<sub>2</sub>SO<sub>4</sub> : plates.

N-Tetra-acetyl : needles from AcOH. M.p. 245°.

Nietzki, Hagenbach, *Ber.*, 1897, **30**, 539.

Borsche, *Ber.*, 1923, **56**, 1939.

## 1 : 2 : 4 : 5-Tetra-aminobenzene.

Free base oxidises very readily in air.

B,4HCl : prisms. Sol. H<sub>2</sub>O. Spar. sol. HCl.

B,H<sub>2</sub>SO<sub>4</sub> : needles. Mod. sol. H<sub>2</sub>O.

B,3H<sub>2</sub>SO<sub>4</sub> : plates. Spar. sol. H<sub>2</sub>O.

2 : 4-N-Diphenyl : C<sub>18</sub>H<sub>18</sub>N<sub>4</sub>. MW, 290.  
Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 207°.

1 : 2 : 4 : 5-N-Tetraphenyl : C<sub>30</sub>H<sub>26</sub>N<sub>4</sub>. MW, 442. Needles. M.p. 173-4°.

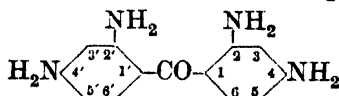
1 : 2 : 4 : 5-N-Tetra-acetyl : needles from AcOH. M.p. 285°.

Nietzki, Hagenbach, *Ber.*, 1887, **20**, 334.

Nietzki, Müller, *Ber.*, 1889, **22**, 440.

Hewitt, Stevenson, *Ber.*, 1898, **31**, 1791.

## 2 : 4 : 2' : 4'-Tetra-aminobenzophenone

C<sub>13</sub>H<sub>14</sub>ON<sub>4</sub> MW, 242

Yellow prisms from EtOH. M.p. 202°. Evolves NH<sub>3</sub> above m.p. Sol. MeOH, Me<sub>2</sub>CO, AcOEt. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

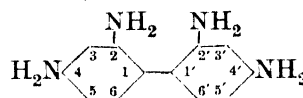
Gulland, Robinson, *J. Chem. Soc.*, 1925, 1499.

## 3 : 4 : 3' : 4'-Tetra-aminobenzophenone.

Yellow needles from H<sub>2</sub>O. M.p. 217°.

Montagne, *Ber.*, 1915, **48**, 1034.

## 2 : 4 : 2' : 4'-Tetra-aminodiphenyl (2 : 2'-Diaminobenzidine)

C<sub>12</sub>H<sub>14</sub>N<sub>4</sub> MW, 214

Leaflets from EtOH.Aq. M.p. 166°. 20% HCl or H<sub>2</sub>SO<sub>4</sub> at 200° → 2 : 7-diaminocarbazole.

2 : 2'-N-Tetra-Me : C<sub>16</sub>H<sub>22</sub>N<sub>4</sub>. MW, 270. Needles from EtOH. M.p. 165.5-166°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH. Spar. sol. H<sub>2</sub>O, ligroin. 4 : 4'-N-Tetra-Me : plates from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 166°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O, ligroin. Insol. H<sub>2</sub>O.

2 : 4 : 2' : 4'-N-Tetra-acetyl : needles from H<sub>2</sub>O. M.p. 285°.

Täuber, *Ber.*, 1890, **23**, 797.

Elbs, Wohlfahrt, *J. prakt. Chem.*, 1902, **66**, 561.

Noelting, Fourniaux, *Ber.*, 1897, **30**, 2940.

Ullmann, Dieterle, *Ber.*, 1904, **37**, 33.

## 2 : 5 : 2' : 5'-Tetra-aminodiphenyl.

Needles from toluene. M.p. 168°. Sol. H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. HCl at 190° → 3 : 6-diaminocarbazole.

Täuber, *Ber.*, 1892, **25**, 130.

## 3 : 4 : 3' : 4'-Tetra-aminodiphenyl (3 : 3'-Diaminobenzidine).

Plates. Rapidly blackens in air.

B,4HCl : needles + 2H<sub>2</sub>O. Sol. H<sub>2</sub>O.

B,H<sub>2</sub>SO<sub>4</sub> : needles. Sol. hot H<sub>2</sub>O. Spar. sol. EtOH, Et<sub>2</sub>O.

4 : 4'-N-Tetra-Me : plates from EtOH. M.p. 168°. Sol. EtOH. Insol. H<sub>2</sub>O. FeCl<sub>3</sub> + HCl → violet col.

Brunner, Witt, *Ber.*, 1887, **20**, 1025.

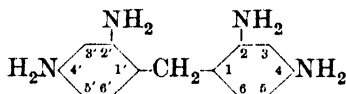
Michler, Pattinson, *Ber.*, 1881, **14**, 2164; 1884, **17**, 118.

**2 : 4 : 2' : 4'-Tetra-aminodiphenylmethane**

670

**2 : 3 : 9 : 10-Tetrabromoanthracene**

**2 : 4 : 2' : 4'-Tetra-aminodiphenylmethane**



$C_{13}H_{16}N_4$  MW, 228

Cryst. from  $H_2O$ . M.p.  $161^\circ$ . Sol. EtOH. Mod. sol.  $H_2O$ . Spar. sol.  $C_6H_6$ . Insol.  $Et_2O$ .

4-N-Di-Me :  $C_{15}H_{20}N_4$ . MW, 256. Cryst. from toluene. M.p.  $188-90^\circ$ . Sol. EtOH,  $CHCl_3$ . Insol.  $H_2O$ .

4-N-Me-4'-N-Di-Me :  $C_{16}H_{22}N_4$ . MW, 270. Cryst. from toluene. M.p.  $95^\circ$ . Sol. EtOH,  $CHCl_3$ . Insol.  $H_2O$ .

4 : 4'-N-Tetra-Me :  $C_{17}H_{24}N_4$ . MW, 284. Needles from EtOH. M.p.  $142^\circ$ . Sol.  $CHCl_3$ ,  $C_6H_6$ , hot EtOH. Mod. sol.  $Et_2O$ . Insol.  $H_2O$ . 4 : 4'-N-Diacetyl : needles from EtOH. M.p.  $244^\circ$ .

2 : 4 : 2' : 4'-N-Tetra-acetyl : cryst. from  $H_2O$ . Mod. sol. EtOH. Spar. sol.  $H_2O$ .

2 : 4 : 2' : 4'-N-Tetrabenzoyl : cryst. from EtOH. M.p.  $275^\circ$ .

Staedel, *Ann.*, 1883, **218**, 341.

Duval, *Compt. rend.*, 1906, **142**, 342.

Pinnow, *Ber.*, 1894, **27**, 3163.

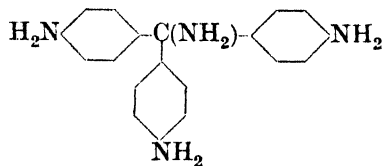
Bayer, D.R.P., 133,709, (*Chem. Zentr.*, 1902, II, 615).

**3 : 4 : 3' : 4'-Tetra-aminodiphenylmethane.**

Plates from  $H_2O$  or  $C_6H_6$ . M.p.  $137-8^\circ$ . Sol. hot  $H_2O$ . Spar. sol. EtOH,  $C_6H_6$ . Insol.  $Et_2O$ ,  $Me_2CO$ .  $FeCl_3 + HCl \rightarrow$  dark red col.

Meyer, Rohmer, *Ber.*, 1900, **33**, 257.

**$\alpha$  : 4 : 4' : 4''-Tetra-aminotriphenylmethane**



$C_{19}H_{20}N_4$  MW, 304

Yellow prisms. Sol. Py. Spar. sol. common org. solvents.

4 : 4' : 4''-N-Hexa-Me :  $C_{25}H_{32}N_4$ . MW, 388. Leaflets from  $C_6H_6$ -ligroin. M.p.  $190.5^\circ$  decomp. Spar. sol.  $Et_2O$ , ligroin.

4 : 4' : 4''-N-Hexa-Et :  $C_{31}H_{44}N_4$ . MW, 472. Needles from ligroin. M.p.  $141.5-142.5^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ . Mod. sol. ligroin.

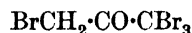
Villiger, Kopetschni, *Ber.*, 1912, **45**, 2920.

Noeltig, Saas, *Ber.*, 1913, **46**, 953.

**2 : 3 : 4 : 6-Tetrabromoacetanilide.**

See under 2 : 3 : 4 : 6-Tetrabromoaniline.

**1 : 1 : 1 : 3-Tetrabromoacetone**



$C_3H_2OBr_4$  MW, 374

Needles. M.p.  $37-8^\circ$ . B.p.  $258^\circ$  decomp.,  $139^\circ/14$  mm.

Tetrahydrate : prisms. M.p.  $62^\circ$ .

Mulder, *J. prakt. Chem.*, 1864, **91**, 475.

de Jong, *Rec. trav. chim.*, 1903, **22**, 286.

Dippy, Watson, Yates, *J. Chem. Soc.*, 1931, 2508.

**1 : 1 : 3 : 3-Tetrabromoacetone**

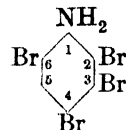


$C_3H_2OBr_4$  MW, 374

Yellow oil. Decomp. on warming. Alkalis  $\rightarrow$  bromoform.

Lederer, D.R.P., 98,009.

**2 : 3 : 4 : 6-Tetrabromoaniline**



$C_6H_3NBr_4$  MW, 409

Cryst. from EtOH. M.p.  $118^\circ$ .

N-Acetyl : 2 : 3 : 4 : 6-tetrabromoacetanilide. Needles from  $C_6H_6$ . M.p.  $228-9^\circ$ .

N-Diacetyl : prisms from pet. ether. M.p.  $164^\circ$ .

$C_6H_3NBr_4$ ,  $C_6H_3(NO_2)_3$ -1 : 3 : 5 : yellow needles. M.p.  $107.5-108^\circ$ .

N-Thionyl : yellow needles from  $C_6H_6$ . M.p.  $78^\circ$ .

Dains, Kenyon, *J. Am. Chem. Soc.*, 1931, **53**, 2363.

Sudborough, Beard, *J. Chem. Soc.*, 1910, **97**, 782.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 50.

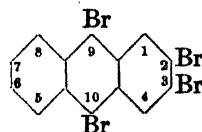
Zincke, Kuchenbecker, *Ann.*, 1904, **330**, 57.

**2 : 3 : 5 : 6-Tetrabromoaniline.**

Needles from EtOH. M.p.  $130^\circ$ .

Claus, *J. prakt. Chem.*, 1895, **51**, 412.

**2 : 3 : 9 : 10-Tetrabromoanthracene**



$C_{14}H_6Br_4$

MW, 494

Yellow needles from  $C_6H_6$ . M.p.  $274^\circ$  ( $265-6^\circ$ ). Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Sublimes. Ox.  $\rightarrow$  2 : 3-dibromoanthraquinone.

Barnett, Cook, *J. Chem. Soc.*, 1935, 1489.  
Meyer, Zahn, *Ann.*, 1913, **396**, 174.

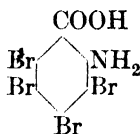
### 2 : 6 : 9 : 10-Tetrabromoanthracene.

Cryst. from  $CCl_4$  or toluene. M.p.  $298-300^\circ$ . Mod. sol. hot toluene. Spar. sol. EtOH,  $C_6H_6$ . Ox.  $\rightarrow$  2 : 6-dibromoanthraquinone.

Grandmougin, *Compt. rend.*, 1921, **173**, 1176.

Kaufler, Imhoff, *Ber.*, 1904, **37**, 4706.

**Tetrabromoanthranilic Acid** (3 : 4 : 5 : 6-Tetrabromo-2-aminobenzoic acid)



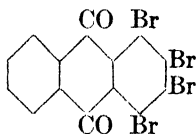
$C_7H_3O_2NBr_4$  MW, 453

Needles from AcOH. M.p.  $204-5^\circ$ . Sol. EtOH, AcOEt. Mod. sol.  $CHCl_3$ , AcOH, xylene.

Lesser, Weiss, *Ber.*, 1913, **46**, 3942.

Grandmougin, *Ber.*, 1914, **47**, 384.

### 1 : 2 : 3 : 4-Tetrabromoanthraquinone



$C_{14}H_4O_2Br_4$  MW, 524

Orange-red needles from ligroin. M.p.  $200-2^\circ$ .

Hofmann, *Monatsh.*, 1915, **36**, 820.

The following tetrabromoanthraquinones of unknown constitution have also been described :

(i) Yellow needles from  $PhNO_2$  or xylene. M.p.  $295^\circ$ .

Bayer, D.R.P., 107,721, (*Chem. Zentr.*, 1900, I, 1176).

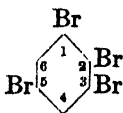
(ii) Yellow needles from  $CS_2$ . M.p. above  $370^\circ$ . Sublimes. NaOH fusion  $\rightarrow$  alizarin.

Hammerschlag, *Ber.*, 1877, **10**, 1213.

(iii) Yellow leaflets from toluene,  $CHCl_3$  or  $CS_2$ . M.p.  $295-300^\circ$ .

Diehl, *Ber.*, 1878, **11**, 182.

### 1 : 2 : 3 : 5-Tetrabromobenzene



$C_6H_2Br_4$

MW, 394

Needles from EtOH. M.p.  $98-5^\circ$ . B.p.  $329^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ . Mod. sol. EtOH.

v. Richter, *Ber.*, 1875, **8**, 1426.

Zincke, Kuchenbecker, *Ann.*, 1904, **330**, 9, 54.

### 1 : 2 : 4 : 5-Tetrabromobenzene.

Cryst. from  $CS_2$ . M.p.  $180-1^\circ$ .  $D^{20}_D$  3.027.

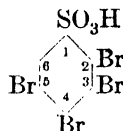
Scheufelen, *Ann.*, 1885, **231**, 187.

Jackson, Gallivan, *Am. Chem. J.*, 1896, **18**, 250.

MacKerrow, *Ber.*, 1891, **24**, 2940.

Zelinsky, *Ber.*, 1901, **34**, 2803.

**2 : 3 : 4 : 5-Tetrabromobenzenesulphonic Acid**



$C_6H_2O_3Br_4S$  MW, 474

Plates from  $H_2O$ . M.p.  $168-9^\circ$ . Sol.  $H_2O$ . Mod. sol. EtOH,  $Et_2O$ .

Chloride :  $C_6HO_2ClBr_4S$ . MW, 492.5. Plates from  $Et_2O$ . M.p.  $120^\circ$ .

Amide :  $C_6H_3O_2NBr_4S$ . MW, 473. Prisms from EtOH. Sinters and darkens at  $240^\circ$  ( $161^\circ$ ).

Spiegelberg, *Ann.*, 1879, **197**, 292.

Lenz, *Ann.*, 1876, **181**, 45.

**2 : 3 : 4 : 6-Tetrabromobenzenesulphonic Acid.**

Needles +  $5H_2O$  from  $H_2O$ . Sol. EtOH,  $H_2O$ .

Chloride : leaflets from  $Et_2O$ . M.p.  $96-5^\circ$ .

Amide : needles from EtOH. M.p. about  $245^\circ$  decomp. Spar. sol.  $H_2O$ .

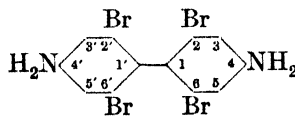
Bässmann, *Ann.*, 1878, **191**, 224.

Beckurts, *Ann.*, 1876, **181**, 216.

Knuth, *Ann.*, 1877, **186**, 299.

### 2 : 6 : 2' : 6'-Tetrabromobenzidine

(2 : 6 : 2' : 6'-Tetrabromo-4 : 4'-diaminodiphenyl)



$C_{12}H_8N_2Br_4$  MW, 500

Cryst. from EtOH.Aq. M.p.  $180^\circ$ . Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ , AcOEt. Spar. sol. ligroin.

4 : 4'-N-Diacetyl : prisms from EtOH. M.p.  $269-70^\circ$ .

Meyer, Meyer, Taeger, *Ber.*, 1920, **53**, 2045.



**3 : 5 : 3' : 5'-Tetrabromobenzidine**

(3 : 5 : 3' : 5'-Tetrabromo-4 : 4'-diaminodiphenyl).  
Needles from xylene. M.p. 288°. Mod. sol.  $C_6H_6$ . Spar. sol. EtOH. Insol.  $H_2O$ ,  $Et_2O$ , acids.

4 : 4'-N-Diacetyl : chars at about 340°.

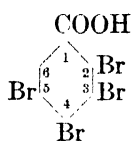
4 : 4'-N-Tetra-acetyl : needles from  $C_6H_6$ .  
M.p. about 306°.

van Roosmalen, *Rec. trav. chim.*, 1934, 53, 359.

Schlenk, *Ann.*, 1908, 363, 335.

Mills, *J. Chem. Soc.*, 1894, 65, 54.

Claus, Risler, *Ber.*, 1881, 14, 86.

**2 : 3 : 4 : 5-Tetrabromobenzoic Acid**

$C_7H_2O_2Br_4$  MW, 438

M.p. 234°. Very sol. EtOH,  $Me_2CO$ . Mod. sol.  $C_6H_6$ . Spar. sol. pet. ether.

Nitrile :  $C_7H_2NBr_4$ . MW, 419. Needles from EtOH. M.p. 124°.

Bunt, *Rec. trav. chim.*, 1929, 48, 121.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, 56, 56.

**2 : 3 : 4 : 6-Tetrabromobenzoic Acid.**

Needles from  $C_6H_6$ . M.p. 179°. Sol. EtOH,  $Et_2O$ . Mod. sol.  $C_6H_6$ . Spar. sol.  $H_2O$ .

Me ester :  $C_8H_4O_2Br_4$ . MW, 452. Needles from EtOH. M.p. 77°.

Et ester :  $C_9H_6O_2Br_4$ . MW, 466. M.p. 31°.

Chloride :  $C_7HOCIBr_4$ . MW, 456.5. Prisms from pet. ether. M.p. 58°. Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ .

Nitrile : needles from EtOH. M.p. 123°. Volatile in steam.

Sudborough, *J. Chem. Soc.*, 1895, 67, 597.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, 56, 52.

Sudborough, Karvé, *J. Indian Inst. Sci.*, 1919, 3, 1.

**2 : 3 : 5 : 6-Tetrabromobenzoic Acid.**

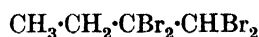
Yellow plates from EtOH. M.p. 295°. Sol.  $C_6H_6$ . Spar. sol. EtOH.

Note.—In the literature this acid is described as of unknown constitution, but since it is different from the other two tetrabromobenzoic acids, it probably possesses the above structure.

Kunckell, Knigge, *Ber.*, 1906, 39, 195.

**Tetrabromo-p-benzoquinone.**

See Bromanil.

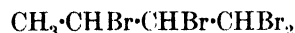
**1 : 1 : 2 : 2-Tetrabromobutane**

$C_4H_6Br_4$  MW, 374

Cryst. Sublimes at 200°.

Bruylants, *Ber.*, 1875, 8, 412.

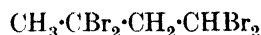
Dupont, *Compt. rend.*, 1909, 148, 1523.

**1 : 1 : 2 : 3-Tetrabromobutane**

$C_4H_6Br_4$  MW, 374

B.p. 148–50°/25 mm.

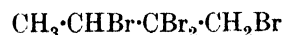
Muskat, Grimsley, *J. Am. Chem. Soc.*, 1933, 55, 2145.

**1 : 1 : 3 : 3-Tetrabromobutane**

$C_4H_6Br_4$  MW, 374

Fuming liq.  $D_4^{16}$  2.7.

Mereshkowsky, *Chem. Zentr.*, 1914, I, 2160.

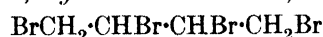
**1 : 2 : 2 : 3-Tetrabromobutane**

$C_4H_6Br_4$  MW, 374

M.p. — 2°. B.p. 97.5°/7 mm.  $D_4^{20}$  2.510.  $n_D^{20}$  1.6070.

Hurd, Meinert, *J. Am. Chem. Soc.*, 1931, 53, 293.

**1 : 2 : 3 : 4-Tetrabromobutane** (*Butadiene tetrabromide, erythrene tetrabromide*)



$C_4H_6Br_4$  MW, 374

Exists in two isomeric forms.

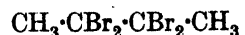
(i) Needles from ligroin. M.p. 118–19°. B.p. 180–1°/60 mm. Spar. sol. ligroin. Volatile in steam.

(ii) Leaflets from pet. ether. M.p. 40–1°. Sol. EtOH,  $Et_2O$ , ligroin.

Perkin, Simonsen, *J. Chem. Soc.*, 1905, 87, 857.

Ciamician, Magnaghi, *Ber.*, 1886, 19, 570; 1887, 20, 3064.

Jacobson, *J. Am. Chem. Soc.*, 1932, 54, 1545.

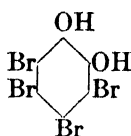
**2 : 2 : 3 : 3-Tetrabromobutane**

$C_4H_6Br_4$  MW, 374

Cryst. from ligroin. M.p. 243°. Sol. ligroin. Insol. EtOH. Dimorphous.

Faworski, *J. prakt. Chem.*, 1890, 42, 144.  
Wislicenus, Schmidt, *Ann.*, 1900, 313, 225.

## Tetrabromocatechol



MW, 426

Needles from EtOH. M.p. 192–3°.  $FeCl_3$  —→ dark blue col.

*Mono-Me ether*: tetrabromoguaiacol.  $C_7H_4O_2Br_4$ . MW, 440. Prisms from EtOH or  $CHCl_3$ . M.p. 162–3°. Sol.  $Et_2O$ ,  $Me_2CO$ . Mod. sol. EtOH,  $CHCl_3$ . Spar. sol. AcOH. Insol.  $H_2O$ .

*Di-Me ether*: tetrabromoveratrol.  $C_8H_6O_2Br_4$ . MW, 454. Cryst. from  $CCl_4$ . M.p. 151–2° (118–20°).

*Methylene ether*: tetrabromomethylenedioxybenzene.  $C_7H_2O_2Br_4$ . MW, 438. Needles from EtOH. M.p. 208–9°.

*Diacetyl*: plates from  $C_6H_6$ . M.p. 215–16°.

*Dibenzoyl*: plates from  $C_6H_6$ -ligroin. M.p. 197–8°.

Frejka, Sefránek, *Chem. Zentr.*, 1936, I, 2338.

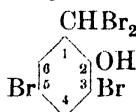
Jackson, Russe, *Am. Chem. J.*, 1906, 35, 169, 178.

Zincke, *Ber.*, 1887, 20, 1778.

Stenhouse, *Ann.*, 1875, 177, 187.

Zetzsche, Sukienik, *Helv. Chim. Acta*, 1927, 10, 101.

$\omega : \omega : 3 : 5$ -Tetrabromo-*o*-cresol (3 : 5-Dibromo-*o*-hydroxybenzylidene bromide)



MW, 424

Needles from pet. ether. M.p. 99°.

*Acetyl*: needles from pet. ether. M.p. 113–14°.

Lindemann, Forth, *Ann.*, 1924, 435, 226.

3 : 4 : 5 : 6-Tetrabromo-*o*-cresol.

Needles from  $CHCl_3$  or AcOH. M.p. 208°. Sol.  $Et_2O$ . Mod. sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. AcOH, ligroin.

*Me ether*:  $C_8H_6OBr_4$ . MW, 438. Needles from AcOH. M.p. 140–5°.

*Acetyl*: needles from EtOH.Aq. M.p. 154°.

Bonneaud, *Bull. soc. chim.*, 1910, 7, 779.

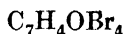
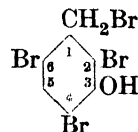
Bodroux, *Compt. rend.*, 1898, 126, 1283.

Zincke, Hedenström, *Ann.*, 1906, 350, 276.

Anselmino, *Ber.*, 1902, 35, 150.

Dict. of Org. Comp.—III.

$\omega : 2 : 4 : 6$ -Tetrabromo-*m*-cresol (2 : 4 : 6-Tribromo-*m*-hydroxybenzyl bromide)



MW, 424

Needles from AcOH. M.p. 149°. Spar. sol. ligroin, pet. ether.

*Acetyl*: needles from AcOH. M.p. 104°.

Auwers, Richter, *Ber.*, 1899, 32, 3382.

2 : 4 : 5 : 6-Tetrabromo-*m*-cresol.

Cryst. from AcOH. M.p. 194°.

*Me ether*:  $C_8H_6OBr_4$ . MW, 438. Needles from EtOH or AcOH. M.p. 145–6°.

*Et ether*:  $C_9H_8OBr_4$ . MW, 452. Needles from  $Et_2O$ . M.p. 108°.

*Acetyl*: needles from ligroin. M.p. 165–6°.

*Benzoyl*: plates from AcOH. M.p. 153–4°.

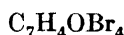
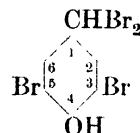
Bureš, Balada, *Chem. Abstracts*, 1928, 22, 3643.

Bonneaud, *Bull. soc. chim.*, 1910, 7, 780.

Anselmino, *Ber.*, 1902, 35, 150.

Auwers, Burrows, *Ber.*, 1899, 32, 3042.

$\omega : \omega : 3 : 5$ -Tetrabromo-*p*-cresol (3 : 5-Dibromo-*p*-hydroxybenzylidene bromide)



MW, 424

Needles from pet. ether. M.p. 98–101.5°. Sol.  $Et_2O$ ,  $C_6H_6$ .

*Me ether*:  $C_8H_6OBr_4$ . MW, 438. Leaflets from ligroin. M.p. 60–4°.

*Acetyl*: leaflets from EtOH. M.p. 80°.

Lindemann, *Ann.*, 1923, 431, 285.

$\omega : 2 : 3 : 5$ -Tetrabromo-*p*-cresol (2 : 3 : 5-Tribromo-*p*-hydroxybenzyl bromide).

Needles from ligroin. M.p. 122°. Sol.  $Et_2O$ , AcOH,  $C_6H_6$ . Mod. sol. ligroin.

*Acetyl*: needles from pet. ether. M.p. 116°.

Zincke, Wiederhold, *Ann.*, 1902, 320, 210.

Auwers, Strecker, *Ann.*, 1904, 334, 330.

2 : 3 : 5 : 6-Tetrabromo-*p*-cresol.

Needles from EtOH. M.p. 198–9°. Sol.  $Et_2O$ ,  $C_6H_6$ , hot AcOH. Mod. sol. EtOH. Spar. sol. ligroin.

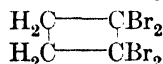
Acetyl : needles from AcOH. M.p. 156°.

Bonneaud, *Bull. soc. chim.*, 1910, **7**, 780.

Zincke, Wiederhold, *Ann.*, 1902, **320**, 207.

Zincke, Buff, *Ann.*, 1905, **341**, 327.

### 1 : 1 : 2 : 2-Tetrabromocyclobutane

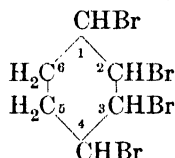


$\text{C}_4\text{H}_4\text{Br}_4$  MW, 372

Prisms from pet. ether. M.p. 126°. Sol. MeOH, EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

Willstätter, Bruce, *Ber.*, 1907, **40**, 3997.

### 1 : 2 : 3 : 4-Tetrabromocyclohexane



$\text{C}_6\text{H}_8\text{Br}_4$  MW, 400

Exists in two forms.

(i) Cryst. from Et<sub>2</sub>O. M.p. 87–8°. NaOEt → bromobenzene.

(ii) Cryst. from MeOH. M.p. 155–6°.

Hofmann, Damm, *Chem. Zentr.*, 1926, **I**, 2343.

Harries, *Ber.*, 1912, **45**, 813.

Bodroux, Taboury, *Compt. rend.*, 1912, **154**, 1514.

Zelinsky, Gorsky, *Ber.*, 1908, **41**, 2483.

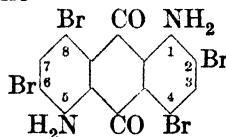
### 1 : 2 : 4 : 5-Tetrabromocyclohexane.

Cryst. from CHCl<sub>3</sub>. M.p. 184–5°.

Baeyer, *Ann.*, 1894, **278**, 96.

Zelinsky, Gorsky, *Ber.*, 1908, **41**, 2481.

### 2 : 4 : 6 : 8-Tetrabromo-1 : 5-diamino-anthraquinone



$\text{C}_{14}\text{H}_6\text{O}_2\text{N}_2\text{Br}_4$  MW, 554

Red needles from PhNO<sub>2</sub>. M.p. about 340°.

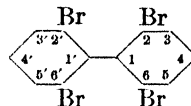
Scholl *et al.*, *Ann.*, 1932, **494**, 221.

### 1 : 3 : 5 : 7-Tetrabromo-2 : 6-diamino-anthraquinone

Yellowish-brown needles from PhNO<sub>2</sub>. Does not melt below 360°.

Badische, D.R.P., 261,270, (*Chem. Zentr.*, 1913, **II**, 194).

### 2 : 6 : 2' : 6'-Tetrabromodiphenyl



$\text{C}_{12}\text{H}_6\text{Br}_4$  MW, 470

Needles from EtOH. M.p. 215°. Mod. sol. common org. solvents.

Meyer, Meyer, Taeger, *Ber.*, 1920, **53**, 2050.

### 3 : 4 : 3' : 4'-Tetrabromodiphenyl.

Cryst. from AcOH. M.p. 169°.

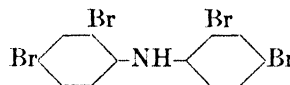
Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

### 3 : 5 : 3' : 5'-Tetrabromodiphenyl.

Cryst. from EtOH. M.p. 186°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH.

See previous reference and also Jacobson, *Ann.*, 1909, **367**, 347.

### 2 : 4 : 2' : 4'-Tetrabromodiphenylamine



$\text{C}_{12}\text{H}_7\text{NBr}_4$  MW, 485

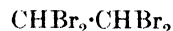
Needles or prisms. M.p. 182°. Sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH. Insol. H<sub>2</sub>O.

N-Me : C<sub>13</sub>H<sub>9</sub>NBr<sub>4</sub>. MW, 499. Prisms. M.p. 142°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH.

Fries, *Ann.*, 1906, **346**, 213.

Hofmann, *Ann.*, 1864, **132**, 166.

### sym.-Tetrabromoethane (Acetylene tetrabromide)



$\text{C}_2\text{H}_2\text{Br}_4$  MW, 346

Liq. B.p. 151°/54 mm., 125°/15 mm., 114°/2 mm. D<sub>4</sub><sup>20</sup> 2.96725. n<sub>D</sub><sup>20</sup> 1.637951. Zn + EtOH → sym.-dibromoethylene.

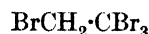
O'Meara, Clemmer, *Chem. Abstracts*, 1929, **23**, 1598.

Lespieau, *Compt. rend.*, 1919, **169**, 31.

Reboul, *Ann.*, 1862, **124**, 269.

Mouneyrat, *Bull. soc. chim.*, 1898, **19**, 498.

### unsym.-Tetrabromoethane



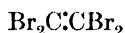
$\text{C}_2\text{H}_2\text{Br}_4$  MW, 346

Liq. B.p. 112.5°/18 mm. D<sub>4</sub><sup>20</sup> 2.87482. n<sub>D</sub><sup>20</sup> 1.627721.

Kaufmann, *Ber.*, 1922, **55**, 258.

Lennox, *Ann.*, 1862, **122**, 124.

Reboul, *Ann.*, 1862, **124**, 270.

**Tetrabromoethylene** (*Perbromoethylene*)

$\text{C}_2\text{Br}_4$  MW, 344

Needles from EtOH. M.p.  $56\cdot5^\circ$ . B.p.  $226\text{--}7^\circ$ ,  $100^\circ/15$  mm. Sublimes. Volatile in steam.

Nekrassow, *Ber.*, 1927, **60**, 1758.

Biltz, *Ber.*, 1902, **35**, 1530.

Anschütz, *Ber.*, 1879, **12**, 2073.

**Tetrabromoguaiacol.**

See under Tetrabromocatechol.

**1 : 2 : 2 : 3-Tetrabromohexane**

$\text{C}_6\text{H}_{10}\text{Br}_4$  MW, 402

B.p.  $130^\circ/3$  mm.  $D^{15}_D$  2.1873.  $n^{15}_D$  1.5850.

Bouis, *Ann. chim.*, 1928, **9**, 402.

**1 : 2 : 3 : 4-Tetrabromohexane** (1 : 3-*Hexadiene tetrabromide*)

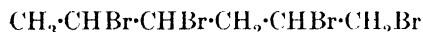


$\text{C}_6\text{H}_{10}\text{Br}_4$  MW, 402

Prisms. M.p.  $91\text{--}2^\circ$ .

Fournier, *Bull. soc. chim.*, 1896, **15**, 403.

Prévost, *Ann. chim.*, 1928, **10**, 176.

**1 : 2 : 4 : 5-Tetrabromohexane**

$\text{C}_6\text{H}_{10}\text{Br}_4$  MW, 402

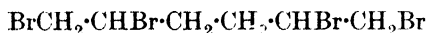
Exists in two forms.

(i) Plates from  $\text{CHCl}_3$ . M.p.  $63\text{--}4^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

(ii) Liq. F.p. below  $-50^\circ$ .

Griner, *Ann. chim. phys.*, 1892, **26**, 336.

**1 : 2 : 5 : 6-Tetrabromohexane** (*Diallyl tetrabromide*)



$\text{C}_6\text{H}_{10}\text{Br}_4$  MW, 402

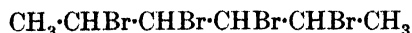
Exists in two stereoisomeric forms.

(i) Rhombic cryst. M.p.  $64\text{--}5^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH,  $\text{C}_6\text{H}_6$ . Mod. sol. ligroin.

(ii) Cryst. M.p.  $53\text{--}4^\circ$ .

Griner, *Ann. chim. phys.*, 1892, **26**, 325.

Ciamician, Anderlini, *Ber.*, 1889, **22**, 2498.

**2 : 3 : 4 : 5-Tetrabromohexane**

$\text{C}_6\text{H}_{10}\text{Br}_4$  MW, 402

Prisms from  $\text{CHCl}_3$ . M.p.  $185^\circ$  ( $182\text{--}3^\circ$ ).

Prévost, *Ann. chim.*, 1928, **10**, 113.

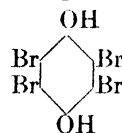
Note.—Two other tetrabromohexanes of unknown constitution have been described :

(i) Plates from AcOH. M.p.  $162\text{--}3^\circ$ .

Merling, *Ann.*, 1891, **264**, 346.

(ii) M.p.  $163\text{--}5^\circ$ .

Mouneyrat, *Ann. chim. phys.*, 1900, **20**, 569.

**Tetrabromohydroquinone**

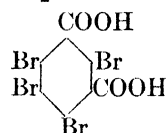
$\text{C}_6\text{H}_2\text{O}_2\text{Br}_4$  MW, 426

Prisms from EtOH- $\text{Et}_2\text{O}$ . M.p.  $244^\circ$ .  $D^{21}$  3.023. Sol. EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .  $\text{FeCl}_3$  in AcOH  $\longrightarrow$  bromanil.

Sarauw, *Ann.*, 1881, **209**, 125.

Bodroux, *Compt. rend.*, 1898, **126**, 1285.

Stenhouse, *Ann.*, 1854, **91**, 310.

**Tetrabromoisophthalic Acid**

$\text{C}_8\text{H}_2\text{O}_4\text{Br}_4$  MW, 482

Needles from  $\text{H}_2\text{O}$ . M.p.  $288\text{--}92^\circ$ . Insol.  $\text{C}_6\text{H}_6$ .

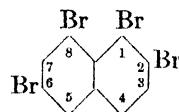
Rupp, *Ber.*, 1896, **29**, 1631.

**Tetrabromomethane.**

See Carbon tetrabromide.

**Tetrabromomethylenedioxybenzene.**

See under Tetrabromocatechol.

**1 : 2 : 6 : 8-Tetrabromonaphthalene**

$\text{C}_{10}\text{H}_4\text{Br}_4$  MW, 444

Needles from  $\text{C}_6\text{H}_6$ . Does not melt below  $315^\circ$ .

Dhar, *J. Chem. Soc.*, 1920, **117**, 993.

**1 : 3 : 5 : 8-Tetrabromonaphthalene.**

Needles from  $\text{C}_6\text{H}_6$ . M.p.  $310^\circ$ .

See previous reference.

**1 : 3 : 6 : 8-Tetrabromonaphthalene.**

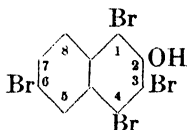
Needles from  $\text{C}_6\text{H}_6$ . Does not melt below  $315^\circ$ .

See previous reference.

**1 : 4 : 6 : 7-Tetrabromonaphthalene.**

Needles from EtOH. M.p. 175°. Sublimes in plates. Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Very spar. sol.  $\text{Et}_2\text{O}$ .

Guareschi, *Gazz. chim. ital.*, 1886, 16, 146.

**1 : 3 : 4 : 6-Tetrabromo-2-naphthol**

$\text{C}_{10}\text{H}_4\text{OBr}_4$  MW, 460

Needles from  $\text{C}_6\text{H}_6$ . M.p. 173–4°. Spar. sol. AcOH. Ox.  $\rightarrow$  4-bromophthalic acid.

*Me ether*:  $\text{C}_{11}\text{H}_6\text{OBr}_4$ . MW, 474. Needles from pet. ether. M.p. 149°.

*Acetyl*: needles from  $\text{C}_6\text{H}_6$ -AcOH. M.p. 192°.

Franzen, Stäuble, *J. prakt. Chem.*, 1922, 103, 377.

Armstrong, Rossiter, *Chem. News*, 1891, 63, 295.

**1 : 3 : 5 : 6-Tetrabromo-2-naphthol.**

Needles from  $\text{CHCl}_3$  or AcOH. M.p. 186°. Sol.  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, pet. ether.

*Acetyl*: needles from  $\text{Me}_2\text{CO}$ . M.p. 156°.

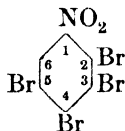
Fries, Schimmelschmidt, *Ann.*, 1930, 484, 280.

**1 : 3 : 6 : 7-Tetrabromo-2-naphthol.**

Needles from AcOH or  $\text{C}_6\text{H}_6$ . M.p. 174°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ .

*Acetyl*: needles from EtOH or AcOH. M.p. 221°.

See previous reference.

**2 : 3 : 4 : 5-Tetrabromonitrobenzene**

$\text{C}_6\text{H}_2\text{O}_2\text{NBr}_4$  MW, 439.

Needles from EtOH. M.p. 107°. Red.  $\rightarrow$  2 : 3 : 4 : 5-tetrabromoaniline.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, 56, 57.

**2 : 3 : 4 : 6-Tetrabromonitrobenzene.**

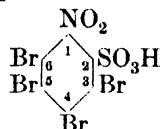
Needles from EtOH. M.p. 96°.

v. Richter, *Ber.*, 1875, 8, 1427.

**2 : 3 : 5 : 6-Tetrabromonitrobenzene.**

Leaflets from EtOH. M.p. 168°.

Claus, *J. prakt. Chem.*, 1895, 51, 412.

**3 : 4 : 5 : 6-Tetrabromonitrobenzene-2-sulphonic Acid**

$\text{C}_6\text{HO}_5\text{NBr}_4\text{S}$

MW, 519

Needles +  $1\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd. 171–3°.

*Chloride*:  $\text{C}_6\text{O}_4\text{NClBr}_3\text{S}$ . MW, 537.5. Prisms from  $\text{Et}_2\text{O}$ . M.p. 172–3°.

*Amide*:  $\text{C}_6\text{H}_2\text{O}_4\text{N}_2\text{Br}_4\text{S}$ . MW, 518. Leaflets. Darkens at 260°, melting with decomp. at higher temp.

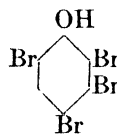
Spiegelberg, *Ann.*, 1878, 197, 297.

**2 : 4 : 5 : 6-Tetrabromonitrobenzene-3-sulphonic Acid.**

Needles. Easily sol.  $\text{H}_2\text{O}$ , EtOH. Conc. HCl in  $\text{H}_2\text{SO}_4$  at 200°  $\rightarrow$  2 : 3 : 4 : 6-tetrabromonitrobenzene.

*Chloride*: plates from  $\text{Et}_2\text{O}$ . M.p. 147–5°.

Langfurth, *Ann.*, 1878, 191, 202.

**2 : 3 : 4 : 6-Tetrabromophenol**

$\text{C}_6\text{H}_2\text{OBr}_4$

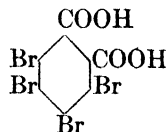
MW, 410

Needles from EtOH. M.p. 113° (120°). Very sol. EtOH. Sublimes.

*Acetyl*: leaflets from AcOH.Aq. M.p. 104–5°.

Hodgson, Walker, Nixon, *J. Chem. Soc.*, 1933, 1054.

Körner, *Ann.*, 1866, 137, 210.

**Tetrabromophthalic Acid**

$\text{C}_8\text{H}_2\text{O}_4\text{Br}_4$

MW, 482

Needles from  $\text{H}_2\text{O}$ . M.p. 266°  $\rightarrow$  anhydride.

*Mono-Me ester*:  $\text{C}_9\text{H}_4\text{O}_4\text{Br}_4$ . MW, 496. Cryst. from MeOH.Aq. M.p. 267°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

*Anhydride*:  $\text{C}_8\text{O}_3\text{Br}_4$ . MW, 464. Cryst. from AcOH-xylene. M.p. 279.5–280.5°.

*Imide*:  $\text{C}_8\text{HO}_2\text{NBr}_4$ . MW, 463. Yellow cryst. from AcOH. Does not melt below 380°. Spar. sol.  $\text{C}_6\text{H}_6$ , toluene, AcOH.

*Anil*: plates from AcOH. M.p. 279–80°.

*o*-Nitroanil: needles from AcOH. M.p. 298–298.5°.

*m*-Nitroanil: plates from xylene. M.p. 301.5–303°.

*p*-Nitroanil: needles from xylene. M.p. 331–331.5°.

*o*-Tolil: plates from C<sub>6</sub>H<sub>6</sub> or AcOH. M.p. 291–3°.

*m*-Tolil: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 273.5–274.5°.

*p*-Tolil: yellow needles from C<sub>6</sub>H<sub>6</sub>. M.p. 280–280.5°.

$\alpha$ -Naphthylimide: yellow needles from C<sub>6</sub>H<sub>6</sub>. M.p. 309–309.5°.

$\beta$ -Naphthylimide: greenish-yellow plates. M.p. 305.5–308°.

Pratt, Young, *J. Am. Chem. Soc.*, 1918, 40, 1416.

Hofmann, *Monatsh.*, 1915, 36, 818.

### 1 : 1 : 1 : 2-Tetrabromopropane

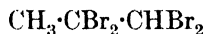


C<sub>3</sub>H<sub>4</sub>Br<sub>4</sub> MW, 360

B.p. 122°/15 mm. D<sub>4</sub><sup>20</sup> 2.679. n<sub>D</sub><sup>20</sup> 1.6187.

Bachman, *J. Am. Chem. Soc.*, 1935, 57, 1090.

### 1 : 1 : 2 : 2-Tetrabromopropane (*Allylene tetrabromide*)



C<sub>3</sub>H<sub>4</sub>Br<sub>4</sub> MW, 360

Liq. B.p. 225–30° part. decomp., 105–7°/9 mm. D<sub>4</sub><sup>20</sup> 2.687. n<sub>D</sub><sup>20</sup> 1.6166.

Hurd, Meinert, Spence, *J. Am. Chem. Soc.*, 1930, 52, 1142.

Oppenheim, *Ann.*, 1864, 132, 124.

### 1 : 1 : 2 : 3-Tetrabromopropane



C<sub>3</sub>H<sub>4</sub>Br<sub>4</sub> MW, 360

B.p. 179–80°/80 mm., 138–40°/17 mm. D<sub>4</sub><sup>0</sup> 2.76.

Mouneyrat, *Compt. rend.*, 1898, 127, 276.

Lespieau, *Ann. chim. phys.*, 1897, 11, 253.

### 1 : 1 : 3 : 3-Tetrabromopropane



C<sub>3</sub>H<sub>4</sub>Br<sub>4</sub> MW, 360

B.p. 154–6°/19 mm. D<sub>4</sub><sup>0</sup> 2.7405, D<sub>4</sub><sup>21</sup> 2.702. n<sub>D</sub><sup>21</sup> 1.6225.

Demjanow, Dojarenko, *Ber.*, 1923, 56, 2202.

### 1 : 2 : 2 : 3-Tetrabromopropane (*Allene tetrabromide*)



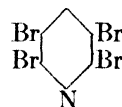
C<sub>3</sub>H<sub>4</sub>Br<sub>4</sub> MW, 360

Colourless liq. with odour resembling camphor. M.p. 10–11°. B.p. 215–30° decomp., 169–70°/80 mm., 115.5°/9 mm. D<sub>4</sub><sup>20</sup> 2.703. n<sub>D</sub><sup>20</sup> 1.6200. Zn dust in EtOH → allene.

Gustavson, Demjanow, *J. prakt. Chem.*, 1888, 38, 204.

Hurd, Meinert, Spence, *J. Am. Chem. Soc.*, 1930, 52, 1143.

### 2 : 3 : 5 : 6-Tetrabromopyridine

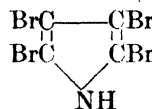


C<sub>5</sub>HNBBr<sub>4</sub> MW, 395

Needles from 50% AcOH. M.p. 103.5–104°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, 51, 940.

### 2 : 3 : 4 : 5-Tetrabromopyrrole



C<sub>4</sub>HNBBr<sub>4</sub> MW, 383

Needles from EtOH. Does not melt below 250°. Sol. EtOH. Spar. sol. H<sub>2</sub>O. KI in EtOH → 2 : 3 : 4 : 5-tetraiodopyrrole.

*N*-Me: C<sub>5</sub>H<sub>3</sub>NBr<sub>4</sub>. MW, 397. Needles from pet. ether. M.p. 154–5° → a blue liq.

*N*-Et: C<sub>6</sub>H<sub>5</sub>NBr<sub>4</sub>. MW, 411. Needles from EtOH. M.p. 90° (83°). Spar. sol. EtOH. Insol. H<sub>2</sub>O.

Plancher, Soncini, *Gazz. chim. ital.*, 1902, 32, ii, 465.

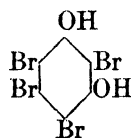
Zanetti, *Ber.*, 1889, 22, 2515.

de Varda, *Ber.*, 1888, 21, 2871.

### Tetrabromoquinone.

See Bromanil.

### Tetrabromoresorcinol



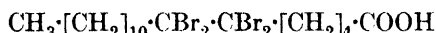
C<sub>6</sub>H<sub>2</sub>O<sub>2</sub>Br<sub>4</sub> MW, 426

Needles from EtOH.Aq. M.p. 167° (163°). Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, hot EtOH. Spar. sol. H<sub>2</sub>O.

*Diacetyl*: m.p. 114° (169°).

Claassen, *Ber.*, 1878, 11, 1440.

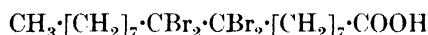
Benedikt, *Monatsh.*, 1880, 1, 366.

**5 : 5 : 6 : 6-Tetrabromostearic Acid** (*Tariric acid tetrabromide*) $\text{C}_{18}\text{H}_{32}\text{O}_2\text{Br}_4$ 

MW, 600

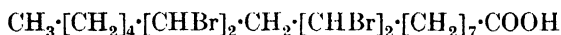
M.p. 125°.

Arnaud, *Compt. rend.*, 1902, **134**, 842;  
*Bull. soc. chim.*, 1892, **7**, 234.

**8 : 8 : 9 : 9-Tetrabromostearic Acid** (*Stearolic acid tetrabromide*) $\text{C}_{18}\text{H}_{32}\text{O}_2\text{Br}_4$ 

MW, 600

Cryst. from EtOH. M.p. 70°.

Overbeck, *Ann.*, 1866, **140**, 56.**8 : 9 : 11 : 12-Tetrabromostearic Acid** (*Linoleic acid tetrabromide*) $\text{C}_{18}\text{H}_{32}\text{O}_2\text{Br}_4$ 

MW, 600

Cryst. from Et<sub>2</sub>O-pet. ether. M.p. 116-17°  
 (112.3-114.3°). Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH,  
 C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. Sn + HCl in EtOH →  
 linoleic acid.

*K salt*: cryst. from EtOH. M.p. 171° (154.7-  
 158.8°).

*Na salt*: cryst. from EtOH. M.p. 194.2-  
 201.1°.

*Ca salt*: cryst. from EtOH. M.p. 208.7-  
 213.4°.

*Sr salt*: cryst. from EtOH. M.p. 200.4-206°.

*Ba salt*: cryst. from EtOH. M.p. 196.3-  
 206.5°.

*Me ester*: C<sub>19</sub>H<sub>34</sub>O<sub>2</sub>Br<sub>4</sub>. MW, 614. M.p. 63°  
 (50-6°).

*Et ester*: C<sub>20</sub>H<sub>36</sub>O<sub>2</sub>Br<sub>4</sub>. MW, 628. Needles.  
 M.p. 63° (58-58.5°).

*Propyl ester*: C<sub>21</sub>H<sub>38</sub>O<sub>2</sub>Br<sub>4</sub>. MW, 642. Cryst.  
 from MeOH. M.p. 45-50°.

*Isopropyl ester*: m.p. 50-2°.

*Allyl ester*: C<sub>21</sub>H<sub>36</sub>O<sub>2</sub>Br<sub>4</sub>. MW, 640. M.p.  
 72-80°.

Smit, *Rec. trav. chim.*, 1930, **49**, 539.

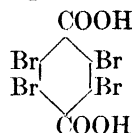
Santos, West, *Chem. Abstracts*, 1928, **22**,  
 761.

Oreta, West, *Chem. Abstracts*, 1927, **21**,  
 3889.

Hazura, *Monatsh.*, 1887, **8**, 149.

Rollet, *Z. physiol. Chem.*, 1909, **62**, 414.

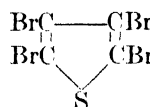
Palmer, Wright, *Ind. Eng. Chem.*, 1914,  
**6**, 822.

**Tetrabromoterephthalic Acid** $\text{C}_8\text{H}_2\text{O}_4\text{Br}_4$ 

MW, 482

Needles from H<sub>2</sub>O. M.p. about 300° decomp.  
 Very spar. sol. EtOH, Et<sub>2</sub>O, AcOH. Insol.  
 C<sub>6</sub>H<sub>6</sub>, cold H<sub>2</sub>O.

Rupp, *Ber.*, 1896, **29**, 1626.

**Tetrabromothiophene** $\text{C}_4\text{Br}_4\text{S}$ 

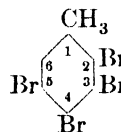
MW, 400

Needles from EtOH. M.p. 117-18°. B.p.  
 326°, 170-3°/13 mm. Fuming HNO<sub>3</sub> → di-  
 bromomaleic acid.

Steinkopf, Jacob, Penz, *Ann.*, 1934,  
**512**, 149.

**α : 2 : 4 : 6-Tetrabromotoluene.**

See 2 : 4 : 6-Tribromobenzyl bromide.

**2 : 3 : 4 : 5-Tetrabromotoluene** $\text{C}_7\text{H}_4\text{Br}_4$ 

MW, 408

Needles. M.p. 111-111.5°.

Nevile, Winther, *Ber.*, 1880, **13**, 976.

**2 : 3 : 4 : 6-Tetrabromotoluene.**

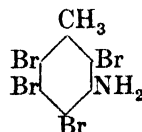
M. p. 105-8°.

Nevile, Winther, *Ber.*, 1880, **13**, 975.

**2 : 3 : 5 : 6-Tetrabromotoluene.**

Needles. M.p. 116-17°. Spar. sol. EtOH.

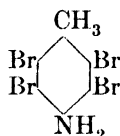
See previous reference.

**2 : 4 : 5 : 6-Tetrabromo-*m*-toluidine** $\text{C}_7\text{H}_5\text{NBr}_4$ 

MW, 423

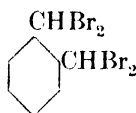
M.p. 223-4°. Mod. sol. EtOH.

See previous reference.

2 : 3 : 5 : 6-Tetrabromo-*p*-toluidine $C_7H_5NBr_4$ 

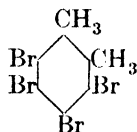
MW, 423

Needles. M.p. 226–7°.

Neville, Winther, *Ber.*, 1881, **14**, 418.Scheufelen, *Ann.*, 1885, **231**, 179.**Tetrabromoveratrol.***See under* 'Tetrabromocatechol. $\omega : \omega : \omega' : \omega'$ -Tetrabromo-*o*-xylene $C_8H_6Br_4$ 

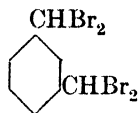
MW, 422

Cryst. from EtOH. M.p. 116°. Sol.  $CHCl_3$ . Somewhat difficultly sol. EtOH. Insol. ligroin. K oxalate in EtOH.Aq.  $\rightarrow$  *o*-phthalaldehyde.

Gabriel, Müller, *Ber.*, 1895, **28**, 1830.Thiele, Günther, *Ann.*, 1906, **347**, 107.3 : 4 : 5 : 6-Tetrabromo-*o*-xylene $C_8H_6Br_4$ 

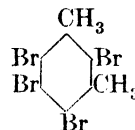
MW, 422

Needles from  $C_6H_6$ . M.p. 262° (254–5°). B.p. 374–5°. Sol. hot  $C_6H_6$ . Very spar. sol. hot EtOH. Dil.  $HNO_3$  + Br at 170°  $\rightarrow$  tetrabromophthalic acid.

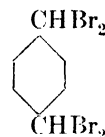
Blümlein, *Ber.*, 1884, **17**, 2492.Klages, Sommer, *Ber.*, 1906, **39**, 2312. $\omega : \omega : \omega' : \omega'$ -Tetrabromo-*m*-xylene $C_8H_6Br_4$ 

MW, 422

Needles from EtOH. M.p. 107°. Sol.  $CHCl_3$ , AcOH,  $C_6H_6$ , ligroin. K oxalate in EtOH  $\rightarrow$  isophthalaldehyde.

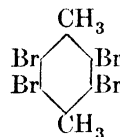
Thiele, Günther, *Ann.*, 1906, **347**, 109.2 : 4 : 5 : 6-Tetrabromo-*m*-xylene $C_8H_6Br_4$ 

MW, 422

M.p. 247° (241°). Sol.  $C_6H_6$ . Insol. EtOH.Datta, Chatterjee, *J. Am. Chem. Soc.*, 1916, **38**, 2550. $\omega : \omega : \omega' : \omega'$ -Tetrabromo-*p*-xylene $C_8H_6Br_4$ 

MW, 422

Prisms from  $CHCl_3$ . M.p. 169°. Sol.  $C_6H_6$ . Spar. sol. EtOH. Hot. conc.  $H_2SO_4 \rightarrow$  terephthalaldehyde + terephthalaldehydic acid.

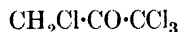
Hönig, *Monatsh.*, 1888, **9**, 1150.Thiele, Günther, *Ann.*, 1906, **347**, 110.2 : 3 : 5 : 6-Tetrabromo-*p*-xylene $C_8H_6Br_4$ 

MW, 422

Needles from toluene. M.p. 256–7° (253°). B.p. 355°. Very spar. sol. hot  $H_2O$ .

Zelinsky, Lepeschkin, *Chem. Zentr.*, 1913, II, 2126.Jacobsen, *Ber.*, 1885, **18**, 359.**Tetrabutylammonium iodide.***See under* Tributylamine.**Tetracarboxytetramethylmethane.***See* Methane-tetracetic Acid.**Tetrachloroacetanilide.***See under* Tetrachloroaniline.

## 1 : 1 : 1 : 3-Tetrachloroacetone

 $C_3H_2OCl_4$ 

MW, 196

Liq. with acid odour which affects mucous membrane. B.p. 183°.  $D_4^{15}$  1.624.  $n_D^{15}$  1.497. Forms hydrate.  $NH_3 \rightarrow CHCl_3$  and chloroacetamide.

Hydrate :  $C_3H_2OCl_4 \cdot 4H_2O$ . MW, 268. Prisms M.p. 46°.

Brochet, *Compt. rend.*, 1894, **119**, 1271.



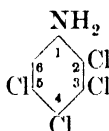
**1 : 1 : 3 : 3-Tetrachloroacetone**

MW, 196

Liq. with strong acrid odour. B.p. 180–2°/718 mm. Forms cryst. hydrate. Forms bisulphite comp.  $\text{Zn} + \text{H}_2\text{SO}_4 \longrightarrow$  acetone.

Hydrate:  $\text{C}_3\text{H}_2\text{OCl}_4 \cdot 4\text{H}_2\text{O}$ . MW, 268. Plates. M.p. 48–9°.

Zincke, Kegel, *Ber.*, 1889, **22**, 1478.

**2 : 3 : 4 : 5-Tetrachloroaniline**

MW, 231

Needles from EtOH. M.p. 118–20°. Very sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

N-Acetyl: 2 : 3 : 4 : 5-tetrachloroacetanilide.  $\text{C}_8\text{H}_5\text{ONCl}_4$ . MW, 273. Cryst. M.p. 160–2°. Very sol. EtOH, Et<sub>2</sub>O, AcOH.

Grandmougin, Seyder, *Ber.*, 1914, **47**, 2369.

Beilstein, Kurbatow, *Ann.*, 1879, **196**, 237.

**2 : 3 : 5 : 6-Tetrachloroaniline.**

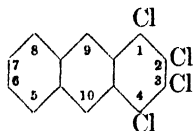
Clusters of needles from ligroin. M.p. 90°. Very sol. EtOH, CS<sub>2</sub>, ligroin.

N-Acetyl: 2 : 3 : 5 : 6-tetrachloroacetanilide. Needles. M.p. 181°. Very sol. EtOH. Spar. sol. AcOH.Aq. N-Me: m.p. 96–7°. N-Et: m.p. 73–4°.

Dyson, George, Hunter, *J. Chem. Soc.*, 1926, 3044.

**Tetrachloroanisole.**

See under Tetrachlorophenol.

**1 : 2 : 3 : 4-Tetrachloroanthracene**

MW, 316

Needles from EtOH-CHCl<sub>3</sub>. M.p. 148–9°. Very sol. CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. hot AcOH. Spar. sol. EtOH, Et<sub>2</sub>O. Ox.  $\longrightarrow$  1 : 2 : 3 : 4-tetrachloroanthraquinone.

Kircher, *Ann.*, 1887, **238**, 346.

**1 : 3 : 9 : 10-Tetrachloroanthracene.**

Yellow needles from AcOH. M.p. 164°. Sol. boiling AcOH. Spar. sol. EtOH. Ox.  $\longrightarrow$  1 : 3-dichloroanthraquinone.

Höchst, D.R.P., 282,818, (*Chem. Zentr.*, 1915, I, 772).

Meyer, Zahn, *Ann.*, 1913, **396**, 172.

**1 : 4 : 5 : 8-Tetrachloroanthracene.**

M.p. 285–6° (275°).

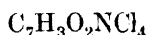
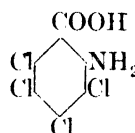
Note.—The positions occupied by the chlorine atoms are not definitely established.

Schilling, *Ber.*, 1913, **46**, 1068.

**2 : 3 : 9 : 10-Tetrachloroanthracene.**

Yellow needles from C<sub>6</sub>H<sub>6</sub>. M.p. 240–1°. Spar. sol. ord. org. solvents except hot C<sub>6</sub>H<sub>6</sub>. Ox.  $\longrightarrow$  2 : 3-dichloroanthraquinone.

Meyer, Zahn, *Ann.*, 1913, **396**, 177.

**Tetrachloroanthranilic Acid**

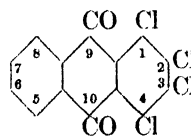
MW, 275

Needles from H<sub>2</sub>O or AcOH. M.p. 182°. Very sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold AcOH, C<sub>6</sub>H<sub>6</sub>. Insol. cold H<sub>2</sub>O. Forms cryst. spar. sol. Ca, Ba salts. Heat above m.p.  $\longrightarrow$  2 : 3 : 4 : 5-tetrachloroaniline.

Me ester:  $\text{C}_8\text{H}_5\text{O}_2\text{NCl}_4$ . MW, 289. Needles from MeOH. M.p. 120–1°.

Badische, D.R.P., 220,839, (*Chem. Zentr.*, 1910, I, 1564).

Villiger, Blangey, *Ber.*, 1909, **42**, 3550.

**1 : 2 : 3 : 4-Tetrachloroanthraquinone**

MW, 346

Yellow needles. M.p. 191°. Very sol. C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. AcOH. Very spar. sol. EtOH, Et<sub>2</sub>O. HNO<sub>3</sub>  $\longrightarrow$  tetrachlorophthalic acid.

Kircher, *Ann.*, 1887, **238**, 345.

**1 : 2 : 5 : 8-Tetrachloroanthraquinone.**

Felted needles from AcOH. M.p. 282–3°.

Goldberg, *J. Chem. Soc.*, 1931, 1792.

**1 : 2 : 6 : 7-Tetrachloroanthraquinone.**

Golden-yellow needles from xylene. M.p. 242°. Sol. cold conc.  $\text{H}_2\text{SO}_4$ .

Barnett, Goodway, Watson, *Ber.*, 1933, **66**, 1885.

**1 : 4 : 5 : 8-Tetrachloroanthraquinone.**

Yellow needles from AcOH or  $\text{PhNO}_2$ . M.p. 341-2°. Sol.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow col.

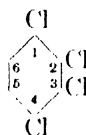
I.C.I., U.S.P., 1,969,044, (*Chem. Abstracts*, 1934, **28**, 6159); E.P., 364,141, (*Chem. Zentr.*, 1933, **I**, 2174).

Goldberg, *J. Chem. Soc.*, 1931, 1792.

**2 : 3 : 6 : 7-Tetrachloroanthraquinone.**

Pale yellow cryst. from *o*-dichlorobenzene. M.p. 348°. Insol. cold. conc.  $\text{H}_2\text{SO}_4$ .

Barnett, Goodway, Watson, *Ber.*, 1933, **66**, 1885.

**1 : 2 : 3 : 4-Tetrachlorobenzene**

$\text{C}_6\text{H}_2\text{Cl}_4$

MW, 216

Needles. M.p. 45-6°. B.p. 254°. Very sol.  $\text{Et}_2\text{O}$ , AcOH,  $\text{CS}_2$ , ligroin. Spar. sol. EtOH.

Cohen, Hartley, *J. Chem. Soc.*, 1905, **87**, 1365.

**1 : 2 : 3 : 5-Tetrachlorobenzene.**

Needles from EtOH. M.p. 51°. B.p. 246°. Very sol.  $\text{CS}_2$ , ligroin. Sol.  $\text{C}_6\text{H}_6$ . Spar. sol. cold EtOH.

Willgerodt, Wilcke, *Ber.*, 1910, **43**, 2752.

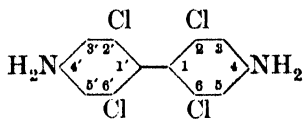
**1 : 2 : 4 : 5-Tetrachlorobenzene.**

Needles from EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{C}_6\text{H}_6^-$  EtOH or  $\text{CS}_2$ . M.p. 139.5-140.5°. B.p. 240°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. boiling EtOH. Insol. cold EtOH.

Qvist, *Chem. Zentr.*, 1934, **II**, 595.

Dow, U.S.P., 1,934,675, (*Chem. Zentr.*, 1934, **I**, 1390).

Pollak, Wienerberger, *Monatsh.*, 1914, **35**, 1472.

**2 : 6 : 2' : 6'-Tetrachlorobenzidine**

$\text{C}_{12}\text{H}_8\text{N}_2\text{Cl}_4$

MW, 322

Cryst. M.p. 212°.

4 : 4'-N-Diacetyl : m.p. 312°.

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

**3 : 5 : 3' : 5'-Tetrachlorobenzidine.**

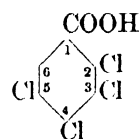
Needles from EtOH-toluene. M.p. 226°. Very sol hot  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ , ligroin.

4 : 4'-N-Diacetyl : m.p. 350°.

Tetra-acetyl : needles from AcOH. M.p. 265-6°.

See previous reference and also

Schlenk, *Ann.*, 1908, **363**, 334.

**2 : 3 : 4 : 5-Tetrachlorobenzoic Acid**

$\text{C}_7\text{H}_2\text{O}_2\text{Cl}_4$

MW, 260

Needles from EtOH. M.p. 186°. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

Et ester :  $\text{C}_9\text{H}_6\text{O}_2\text{Cl}_4$ . MW, 288. Needles from EtOH. M.p. 34-5°.

Nitrile :  $\text{C}_7\text{HNC}_2\text{Cl}_4$ . MW, 241. Needles. M.p. 84°.

Tust, *Ber.*, 1887, **20**, 2439.

**2 : 3 : 4 : 6-Tetrachlorobenzoic Acid.**

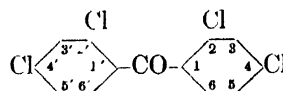
Nitrile : needles. M.p. 81°.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 48, 66.

**2 : 3 : 5 : 6-Tetrachlorobenzoic Acid.**

Nitrile : needles. M.p. 72°.

See previous reference.

**2 : 4 : 2' : 4'-Tetrachlorobenzophenone**

$\text{C}_{13}\text{H}_6\text{OCl}_4$

MW, 320

Cryst. M.p. 78°. Spar. sol. EtOH.Aq.

Cohen, *Rec. trav. chim.*, 1919, **38**, 116.

**2 : 5 : 2' : 4'-Tetrachlorobenzophenone.**

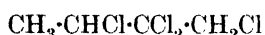
Needles from EtOH. M.p. 176°.

Ganzmüller, *J. prakt. Chem.*, 1933, **138**, 312.

**2 : 5 : 2' : 5'-Tetrachlorobenzophenone.**

Plates from EtOH. M.p. 128°. Very sol. hot EtOH, pet. ether. Spar. sol. cold EtOH.

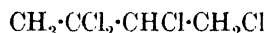
Norris, Green, *Am. Chem. J.*, 1901, **26**, 498.

**1 : 2 : 2 : 3-Tetrachlorobutane****1 : 2 : 2 : 3-Tetrachlorobutane**

$\text{C}_4\text{H}_6\text{Cl}_4$  MW, 196

Liq. B.p. 85°/10 mm.

Garzarolli-Thurnlackh, *Ann.*, 1882, **213**, 372.

**1 : 2 : 3 : 3-Tetrachlorobutane**

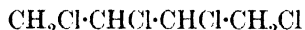
$\text{C}_4\text{H}_6\text{Cl}_4$  MW, 196

Liq. B.p. 90°/32 mm., 55–7°/10 mm.  $D_4^{20}$  1.4204.  $n_D^{20}$  1.4958.

du Pont, U.S.P., 1,964,720, (*Chem. Zentr.*, 1934, II, 3180).

Carothers, Berchet, *J. Am. Chem. Soc.*, 1933, **44**, 1631.

**1 : 2 : 3 : 4-Tetrachlorobutane** (*Butadiene tetrachloride, erythrene tetrachloride*)

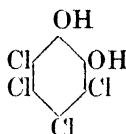


$\text{C}_4\text{H}_6\text{Cl}_4$  MW, 196

Prisms with camphor-like odour from EtOH. M.p. 73–4°. B.p. 130–40°/50 mm.

Backer, Strating, *Rec. trav. chim.*, 1935, **54**, 55.

Henninger, *Ann. chim. phys.*, 1886, **7**, 229.

**Tetrachlorocatechol**

$\text{C}_6\text{H}_2\text{O}_2\text{Cl}_4$  MW, 248

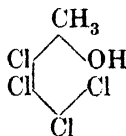
Cryst. from EtOH.Aq. or  $\text{C}_6\text{H}_6$ : cryst. +  $\text{H}_2\text{O}$  from AcOH.Aq. M.p. 194–5°, anhyd. 110°. Cryst. +  $3\text{H}_2\text{O}$  from AcOH.Aq., m.p. 94° Spar. sol.  $\text{C}_6\text{H}_6$ . Ox.  $\rightarrow$  tetrachloro-*o*-benzoquinone.

*Mono-Me ether*: tetrachloroguaiacol.  $\text{C}_7\text{H}_4\text{O}_2\text{Cl}_4$ . MW, 262. Cryst. from hot  $\text{H}_2\text{O}$ . M.p. 185–6°.

*Di-Me ether*: tetrachloroveratrol.  $\text{C}_8\text{H}_6\text{O}_2\text{Cl}_4$ . MW, 276. Needles from AcOH. M.p. 190°.

*Diacetyl*: needles from AcOH. M.p. 190°.

Zincke, Küster, *Ber.*, 1888, **21**, 2729.

**Tetrachloro-*o*-cresol**

$\text{C}_7\text{H}_4\text{OCl}_4$  MW, 246

682

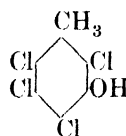
**2 : 4 : 5 : 7-Tetrachloro-1 : 8-diamino-anthraquinone**

Needles from  $\text{C}_6\text{H}_6$ . M.p. 190°. Very sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH,  $\text{C}_6\text{H}_6$ .

*Me ether*:  $\text{C}_8\text{H}_6\text{OCl}_4$ . MW, 260. Needles from MeOH. M.p. 114°.

*Acetyl*: needles from AcOH. M.p. 136°.

Zincke, Pfaffendorf, *Ann.*, 1912, **394**, 12.

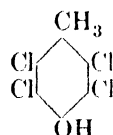
**Tetrachloro-*m*-cresol**

$\text{C}_7\text{H}_4\text{OCl}_4$  MW, 246

Needles from pet. ether. M.p. 189–90°. Sol. ord. org. solvents.

*Acetyl*: needles from AcOH. M.p. 117°.

Crowther, McCombie, *J. Chem. Soc.*, 1913, **103**, 546.

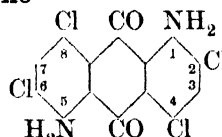
**Tetrachloro-*p*-cresol**

$\text{C}_7\text{H}_4\text{OCl}_4$  MW, 246

Needles from AcOH or  $\text{C}_6\text{H}_6$ -ligroin. M.p. 190°. Very sol. EtOH, AcOH,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin.

*Acetyl*: needles from  $\text{C}_6\text{H}_6$ , plates from AcOH.Aq. M.p. 112°.

Zincke, *Ann.*, 1903, **328**, 281.

**2 : 4 : 6 : 8-Tetrachloro-1 : 5-diamino-anthraquinone**

$\text{C}_{14}\text{H}_6\text{O}_2\text{N}_2\text{Cl}_4$  MW, 376

Brown needles from  $\text{PhNO}_2$ . Spar. sol. cold  $\text{PhNO}_2$ . Insol. EtOH, cold AcOH.  $\text{H}_2\text{SO}_4 \rightarrow$  olive col.

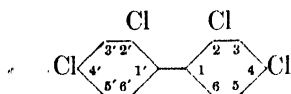
Badische, D.R.P., 158,951, (*Chem. Zentr.*, 1905, I, 842).

**2 : 4 : 5 : 7-Tetrachloro-1 : 8-diamino-anthraquinone.**

Pale brown plates from  $\text{PhNO}_2$ . Sol. hot  $\text{PhNO}_2$ . Spar. sol. EtOH.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow col.

See previous reference.

## 2 : 4 : 2' : 4'-Tetrachlorodiphenyl



$C_{12}H_6Cl_4$  MW, 292

Cryst. from  $C_6H_6$ -ligroin. M.p.  $83^\circ$ . Very sol. warm EtOH,  $C_6H_6$ . Spar. sol. ligroin.

Ullmann, *Ann.*, 1904, **332**, 55.

## 2 : 5 : 2' : 5'-Tetrachlorodiphenyl.

Yellow cryst. from MeOH or pet. ether. M.p.  $84-5^\circ$ .

Meyer, Hofmann, *Monatsh.*, 1917, **38**, 145.

## 2 : 6 : 2' : 6'-Tetrachlorodiphenyl.

Plates. M.p.  $199^\circ$ .

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

## 3 : 4 : 3' : 4'-Tetrachlorodiphenyl.

Colourless needles from AcOH. M.p.  $171^\circ$ . B.p.  $230^\circ/50$  mm. Very sol. EtOH,  $Et_2O$ .

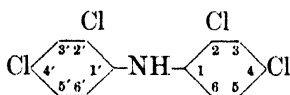
See previous reference and also Cain, *J. Chem. Soc.*, 1904, **85**, 7.

## 3 : 5 : 3' : 5'-Tetrachlorodiphenyl.

M.p.  $162^\circ$ .

Roosmalen, *Rec. trav. chim.*, 1934, **53**, 359.

## 2 : 4 : 2' : 4'-Tetrachlorodiphenylamine



$C_{12}H_7NCl_4$  MW, 307

Prisms or needles. M.p.  $141-2^\circ$  ( $134^\circ$ ). Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ .

N-Me :  $C_{13}H_9NCl_4$ . MW, 321. Prisms. M.p.  $96-7^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ .

Krollpfeiffer, Wolf, Walbrecht, *Ber.*, 1934, **67**, 908.

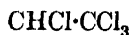
Gnehm, *Ber.*, 1875, **8**, 1040.

## 2 : 3 : 5 : 4'-Tetrachlorodiphenylamine.

Cryst. from EtOH.Aq. M.p.  $107-8^\circ$ . Very sol. EtOH,  $Et_2O$ , ligroin.

Jacobson, *Ann.*, 1909, **367**, 339.

## 1 : 1 : 1 : 2-Tetrachloroethane



$C_2H_2Cl_4$

MW, 168

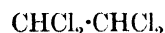
Liq. B.p.  $135.1^\circ$ .  $D_4^{25}$  1.5424.

I.G., D.R.P., 530,649, (*Chem. Zentr.*, 1931, II, 1920).

Prins, *Rec. trav. chim.*, 1926, **45**, 80.

Kokatnur, *J. Am. Chem. Soc.*, 1919, **41**, 123.

**sym.-Tetrachloroethane** (1 : 1 : 2 : 2-Tetrachloroethane, acetylene tetrachloride)



$C_2H_2Cl_4$

MW, 168

Colourless liq. with odour similar to  $CHCl_3$ . F.p.  $-43.8^\circ$ . M.p.  $-36^\circ$ . B.p.  $146.2^\circ$ ,  $62^\circ/45$  mm.,  $55^\circ/17$  mm.  $D_4^{25}$  1.5881,  $D^{15}$  1.60255.  $n_D^{15}$  1.49678. Volatile in steam. Sp. heat 0.286 cal./gm. at  $20^\circ$ . Solvent for P, S, fats, resins, cellulose acetate, rubber, etc. Non-inflammable. Alkalis  $\rightarrow$  trichloroethylene.  $H_2O + Fe$  or  $Al \rightarrow$  sym.-dichloroethylene.  $AlCl_3$  at  $110^\circ \rightarrow$  1 : 1 : 1 : 2-tetrachloroethane. Narcotic and poisonous.

Ruhrchemie A.G., U.S.P., 2,016,658, (*Chem. Abstracts*, 1935, **29**, 8005).

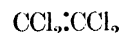
Favorskii, Margules, Davuidova, *Chem. Abstracts*, 1935, **29**, 7271.

Frydlender, *Chem. Abstracts*, 1935, **29**, 7935.

I.G., F.P., 739,183, (*Chem. Zentr.*, 1933, I, 2312).

Timmermans, *Bull. soc. chim. Belg.*, 1927, **36**, 502.

## Tetrachloroethylene (Perchloroethylene)



$C_2Cl_4$

MW, 166.

Liq. F.p.  $-22.35^\circ$ . M.p.  $-19^\circ$ . B.p.  $121.20^\circ$ ,  $33.2^\circ/30$  mm.  $D_4^{20}$  1.65582.  $n_D^{20}$  1.50180. Heat of comb.  $C_p$  162.5 Cal. Sp. heat at  $20^\circ$  0.216 cal./gm.  $Cl \rightarrow$  hexachloroethane. Vapour is poisonous. Extensively used as industrial solvent. Non-inflammable.

Frydlender, *Chem. Abstracts*, 1935, **29**, 7935.

Reilly, U.S.P., 1,947,491, (*Chem. Abstracts*, 1934, **28**, 2371).

Timmermans, Hennault-Roland, *J. chim. phys.*, 1930, **27**, 401.

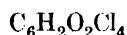
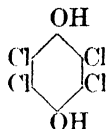
Thusen, U.S.P., 1,590,265, (*Chem. Abstracts*, 1926, **20**, 3015).

Weisen, Wightman, *J. Phys. Chem.*, 1919, **23**, 415.

## Tetrachloroguaiacol.

See under Tetrachlorocatechol.

## Tetrachlorohydroquinone



MW, 248

Needles from AcOH. M.p. 232°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O, CCl<sub>4</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Sublimes.

*Di-Me ether*: C<sub>8</sub>H<sub>8</sub>O<sub>2</sub>Cl<sub>4</sub>. MW, 276. Needles. M.p. 164°. Sol. EtOH, AcOH. Mod. sol. Et<sub>2</sub>O. Sublimes.

*Me-Et ether*: C<sub>9</sub>H<sub>8</sub>O<sub>2</sub>Cl<sub>4</sub>. MW, 290. Needles from EtOH. M.p. 101°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin. Mod. sol. EtOH, AcOH. Sublimes.

*Di-Et ether*: C<sub>10</sub>H<sub>10</sub>O<sub>2</sub>Cl<sub>4</sub>. MW, 304. Needles. M.p. 112°. Sol. EtOH, Et<sub>2</sub>O. Sublimes.

*Diacetyl*: sublimes in needles. M.p. 245°.

*Dipropionyl*: cryst. M.p. 160°.

*Dibutyryl*: cryst. M.p. 137°.

*Dibenzoyl*: needles from CS<sub>2</sub>. M.p. 232°.

Graebe, *Ann.*, 1868, **146**, 20.

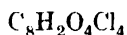
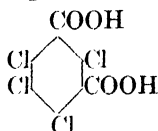
Bouveault, *Compt. rend.*, 1899, **129**, 55.

Fiala, *Monatsh.*, 1885, **6**, 912.

Niemeyer, *Ann.*, 1885, **228**, 324.

Klinger, *Ann.*, 1911, **382**, 221.

## Tetrachloroisophthalic Acid



MW, 304

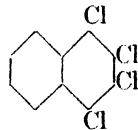
Needles. M.p. 267-9°. Sol. EtOH. Very spar. sol. C<sub>6</sub>H<sub>6</sub>.

Rupp, *Ber.*, 1896, **29**, 1632.

## Tetrachloromethane.

See Carbon tetrachloride.

## 1 : 2 : 3 : 4-Tetrachloronaphthalene



MW, 266

M.p. 198°.

v. Braun *et al.*, *Ber.*, 1923, **56**, 2337.

The following tetrachloronaphthalenes of unknown structure have also been described :

(i) Needles from Et<sub>2</sub>O-ligroin. M.p. 130°.

Faust, Saame, *Ann.*, 1871, **160**, 72.

Widman, *Bull. soc. chim.*, 1877, **28**, 506.

(ii) Needles. M.p. 194°. Spar. sol. EtOH.

Atterberg, *Ber.*, 1876, **9**, 318.

(iii) Needles. M.p. 176°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, AcOH.

Atterberg, Widman, *Bull. soc. chim.*, 1877, **28**, 507.

(iv) Needles. M.p. 141°. Spar. sol. EtOH.

Atterberg, Widman, *Bull. soc. chim.*, 1877, **28**, 507.

(v) Needles. M.p. 180°. Spar. sol. EtOH.

Atterberg, Widman, *Bull. soc. chim.*, 1877, **28**, 514.

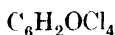
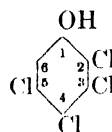
(vi) Needles. M.p. 159-5-160-5°.

Alén, *Bull. soc. chim.*, 1881, **36**, 435.

## Tetrachlorophenetole.

See under Tetrachlorophenol.

## 2 : 3 : 4 : 5-Tetrachlorophenol



MW, 232

Needles from ligroin. M.p. 116-17°.

*Benzoyl*: m.p. 110°.

Holleman, *Rec. trav. chim.*, 1921, **40**, 318.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.

## 2 : 3 : 4 : 6-Tetrachlorophenol.

Needles from ligroin. M.p. 70°. B.p. 150°/15 mm. Sol. EtOH, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin. Mod. sol. pet. ether. Spar. sol. H<sub>2</sub>O.

*Me ether*: 2 : 3 : 4 : 6-tetrachloroanisole. C<sub>7</sub>H<sub>4</sub>OCl<sub>4</sub>. MW, 246. Needles from MeOH. M.p. 64-5°.

*Et ether*: 2 : 3 : 4 : 6-tetrachlorophenetole. C<sub>8</sub>H<sub>6</sub>OCl<sub>4</sub>. MW, 260. Needles from EtOH. M.p. 55°.

*Acetyl*: cryst. from EtOH. M.p. 65-6°.

*Benzoyl*: m.p. 108°.

Lock, *Monatsh.*, 1936, **67**, 320.

Holleman, *Rec. trav. chim.*, 1921, **40**, 318

Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.

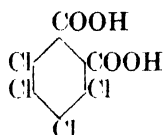
## 2 : 3 : 5 : 6-Tetrachlorophenol.

Leaflets from ligroin. M.p. 115°.

*Benzoyl*: m.p. 136°.

See last two references above.

## Tetrachlorophthalic Acid



$C_8H_2O_4Cl_4$  MW, 304

Plates from  $H_2O$ . At  $98^\circ \rightarrow$  anhydride. Very sol.  $Me_2CO$ . Sol.  $EtOH$ ,  $Et_2O$ . Spar. sol.  $H_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

*Mono-Me ester*:  $C_9H_4O_4Cl_4$ . MW, 318. Needles. M.p.  $142^\circ$ .

*Di-Me ester*:  $C_{10}H_6O_4Cl_4$ . MW, 332. Prisms. M.p.  $92^\circ$ .

*Mono-Et ester*:  $C_{10}H_6O_4Cl_4$ . MW, 332. M.p.  $94-5^\circ$ .

*Di-Et ester*:  $C_{12}H_{10}O_4Cl_4$ . MW, 360. Cryst. M.p.  $60-5^\circ$ .

*Dihexadecyl ester*: plates from  $AcOEt$ . M.p.  $49-50^\circ$ .

*Monobenzyl ester*: m.p.  $130-1^\circ$ .

*Dibenzyl ester*: needles from  $MeOH$ . M.p.  $92-3^\circ$ .

*Di-p-nitrobenzyl ester*: needles from  $C_6H_6$ . M.p.  $180-1^\circ$ .

*sym.-Dichloride*:  $C_8Cl_2Cl_6$ . MW, 341. Prisms from pet. ether. M.p.  $48^\circ$ .

*unsym.-Dichloride*: needles from pet. ether. M.p.  $137^\circ$ .

*Anhydride*:  $C_8O_3Cl_4$ . MW, 286. Needles. M.p.  $255^\circ$ . Sublimes. Spar. sol.  $Et_2O$ .

*Imide*:  $C_8HO_2NCl_4$ . MW, 285. Plates from  $AcOH$ . M.p.  $338-9^\circ$ .

*Anil*: plates from  $AcOH$ . M.p.  $268-9^\circ$ .

*o-Nitroanil*: plates from  $C_6H_6$ . M.p.  $272-3^\circ$ .

*m-Nitroanil*: plates from  $AcOH$ -xylene. M.p.  $300-301-5^\circ$ .

*p-Nitroanil*: needles from  $C_6H_6$ . M.p.  $292-7^\circ$ .

*o-Tolil*: plates from  $AcOH$ . M.p.  $232-236-5^\circ$ .

*m-Tolil*: plates. M.p.  $245-5-246-5^\circ$ .

*p-Tolil*: exists in two forms. (i) Plates from  $AcOH$ . M.p.  $207^\circ$ . (ii) Yellow needles from  $AcOH$ . M.p.  $214-15^\circ$ .

$\beta$ -Naphthylimide: m.p.  $287^\circ$ .

Salkind, Belikova, *Brit. Chem. Abstracts*, 1936, 331.

Kirpal, Kunze, *Ber.*, 1929, 62, 2102.

Pratt, Perkins, *J. Am. Chem. Soc.*, 1918, 40, 203.

Tingle, Bates, *J. Am. Chem. Soc.*, 1910, 32, 1325.

Eckert, Steiner, *Monatsh.*, 1915, 36, 272.

Graebe, *Ann.*, 1887, 238, 332.

Meyer, Jugilewitsch, *Ber.*, 1897, 30, 780.

## 1 : 1 : 1 : 2-Tetrachloropropane

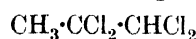


$C_3H_4Cl_4$  MW, 182

Oil. M.p.  $-65^\circ$ . B.p.  $152-3^\circ$ .  $D^{20}$  1.473. Insol.  $H_2O$ .

Henry, *Rec. trav. chim.*, 1905, 24, 333.

## 1 : 1 : 2 : 2-Tetrachloropropane



$C_3H_4Cl_4$  MW, 182

Liq. B.p.  $153^\circ$ .  $D^{13}$  1.47. Misc. with  $EtOH$ ,  $Et_2O$ .

Borsche, Fittig, *Ann.*, 1865, 133, 114.

## 1 : 1 : 2 : 3-Tetrachloropropane



$C_3H_4Cl_4$  MW, 182

Liq. B.p.  $179-80^\circ/756-6$  mm.  $D^{15}$  1.521.

Hartenstein, *J. prakt. Chem.*, 1873, 7, 313.

Mouneyrat, *Bull. soc. chim.*, 1900, 21, 621.

## 1 : 2 : 2 : 3-Tetrachloropropane

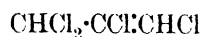


$C_3H_4Cl_4$  MW, 182

Liq. B.p.  $164^\circ$ .  $D^{17}$  1.496.

Herzfelder, *Ber.*, 1893, 26, 2436.

## 1 : 2 : 3 : 3-Tetrachloropropylene

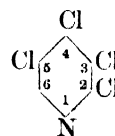


$C_3H_2Cl_4$  MW, 180

B.p.  $165^\circ$ .  $D_4^{24}$  1.5274.  $n_D^{18}$  1.5272.

Prins, *J. prakt. Chem.*, 1914, 89, 421.

## 2 : 3 : 4 : 5-Tetrachloropyridine



$C_5HNCI_4$  MW, 217

Needles from  $EtOH$ . M.p.  $21-2^\circ$ . B.p.  $135-7^\circ/24$  mm. Volatile in steam.

Sell, Dootson, *J. Chem. Soc.*, 1899, 75, 986; 1900, 77, 3.

## 2 : 3 : 4 : 6-Tetrachloropyridine.

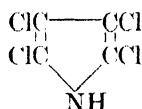
Plates from 50%  $EtOH$ . M.p.  $74-5^\circ$ . B.p.  $130-5^\circ/16-20$  mm. Insol.  $H_2O$ , acids.

Sell, Dootson, *J. Chem. Soc.*, 1898, 73, 440; 1900, 77, 1.

**2 : 3 : 5 : 6-Tetrachloropyridine.**

Cryst. from EtOH.Aq. M.p. 90–1°. B.p. 250–1°, 125–30°/16–20 mm. Sol. EtOH, Et<sub>2</sub>O, pet. ether. Insol. H<sub>2</sub>O, acids.

Sell, Dootson, *J. Chem. Soc.*, 1897, **71**, 1081; 1898, **73**, 439.

**2 : 3 : 4 : 5-Tetrachloropyrrole**C<sub>4</sub>HNC<sub>4</sub>

MW, 205

Plates from pet. ether. M.p. 110°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

*N-Me*: C<sub>5</sub>H<sub>3</sub>NC<sub>4</sub>. MW, 219. Needles from pet. ether. M.p. 118–19°. Spar. sol. H<sub>2</sub>O. Volatile in steam.

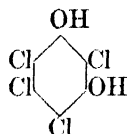
*N-Phenyl*: C<sub>10</sub>H<sub>5</sub>NC<sub>4</sub>. MW, 281. Cryst. from ligroin. M.p. 93°.

Anschütz, Beavis, *Ann.*, 1897, **295**, 30.

Mazzara, Borgo, *Gazz. chim. ital.*, 1904, **34**, i, 258.

Kalle, D.R.P., 38,423, (*Chem. Zentr.*, 1887, 423).

Ciamician, Silber, *Ber.*, 1885, **18**, 1763.

**Tetrachlororesorcinol**C<sub>6</sub>H<sub>2</sub>O<sub>2</sub>Cl<sub>4</sub>

MW, 248

Needles from H<sub>2</sub>O. M.p. 141°. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>. Mod. sol. H<sub>2</sub>O.

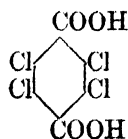
*Di-Et ether*: C<sub>10</sub>H<sub>10</sub>O<sub>2</sub>Cl<sub>4</sub>. MW, 304. Needles from EtOH. M.p. 73°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>, ligroin. Spar. sol. EtOH.

*Dipropyl ether*: C<sub>12</sub>H<sub>14</sub>O<sub>2</sub>Cl<sub>4</sub>. MW, 332. Oil. Decomp. at 100°. Sol. EtOH, Et<sub>2</sub>O, AcOH.

*Diacetyl*: needles. M.p. 145°.

Zincke, Fuchs, *Ber.*, 1892, **25**, 2689.

Jackson, Carlton, *Am. Chem. J.*, 1914, **31**, 379.

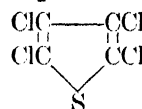
**Tetrachloroterephthalic Acid**C<sub>8</sub>H<sub>2</sub>O<sub>4</sub>Cl<sub>4</sub>

MW, 304

Needles from AcOH. M.p. about 330° (279–81°). Sol. EtOH, Et<sub>2</sub>O, AcOH. Insol. H<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Qvist, Holmberg, *Chem. Zentr.*, 1932, **II**, 2816.

Rupp, *Ber.*, 1896, **29**, 1628.

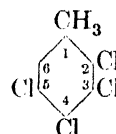
**Tetrachlorothiophene**C<sub>4</sub>Cl<sub>4</sub>S

MW, 222

Cryst. from EtOH.Aq. M.p. 36°. B.p. 240–5°.

Weitz, *Ber.*, 1884, **17**, 795.

Rosenberg, *Ber.*, 1886, **19**, 650.

**2 : 3 : 4 : 5-Tetrachlorotoluene**C<sub>7</sub>H<sub>4</sub>Cl<sub>4</sub>

MW, 230

Needles from MeOH. M.p. 97–8°.

Cohen, Dakin, *J. Chem. Soc.*, 1904, **85**, 1285; 1906, **89**, 1454.

**2 : 3 : 4 : 6-Tetrachlorotoluene.**

Needles from EtOH. M.p. 91.5–92°.

Bureš, Trpisovska, *Chem. Zentr.*, 1936, **I**, 1209.

Cohen, Dakin, *J. Chem. Soc.*, 1904, **85**, 1280.

**2 : 3 : 5 : 6-Tetrachlorotoluene.**

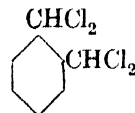
Needles from MeOH. M.p. 93–4°. Sublimes.

Qvist, Holmberg, *Chem. Zentr.*, 1932, **II**, 2816.

Cohen, Dakin, *J. Chem. Soc.*, 1904, **85**, 1281.

**Tetrachloroveratrol.**

See under Tetrachlorocatechol.

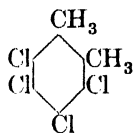
 **$\omega : \omega : \omega' : \omega'$ -Tetrachloro-*o*-xylene**C<sub>8</sub>H<sub>6</sub>Cl<sub>4</sub>

MW, 244.

Cryst. from Et<sub>2</sub>O. M.p. 89°. B.p. 273–4°. D<sub>4</sub><sup>o</sup> 1.601. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

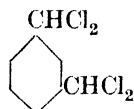
Hjelt, *Ber.*, 1885, **18**, 2879.

Colson, Gautier, *Ann. chim. phys.*, 1887, **11**, 25.

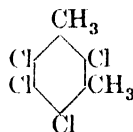
3 : 4 : 5 : 6-Tetrachloro-*o*-xylene $C_8H_6Cl_4$ 

MW, 244

Needles from  $Et_2O$ . M.p.  $215^\circ$ . Sublimes.  
Sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ . Non-volatile in steam.

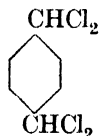
Claus, Kautz, *Ber.*, 1885, **18**, 1369.Datta, Chatterjee, *J. Am. Chem. Soc.*, 1917, **38**, 2549. $\omega : \omega' : \omega' : \omega'$ -Tetrachloro-*m*-xylene $C_8H_6Cl_4$ 

MW, 244

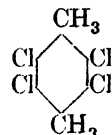
B.p.  $273^\circ$ .Colson, Gautier, *Bull. soc. chim.*, 1886, **45**, 509.2 : 4 : 5 : 6-Tetrachloro-*m*-xylene $C_8H_6Cl_4$ 

MW, 244

Needles from  $EtOH-CHCl_3$ . M.p.  $219^\circ$  ( $212^\circ$ ).  
Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $EtOH$ .

Datta, Fernandes, *J. Am. Chem. Soc.*, 1917, **38**, 1810.Bureš, Borgmann, *Chem. Zentr.*, 1928, **I**, 1171. $\omega : \omega' : \omega' : \omega'$ -Tetrachloro-*p*-xylene $C_8H_6Cl_4$ 

MW, 244

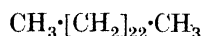
Cryst. from  $Et_2O$ . M.p.  $93^\circ$ .  $D_4^{20}$  1.606.Colson, Gautier, *Ann. chim. phys.*, 1887, **11**, 24.2 : 3 : 5 : 6-Tetrachloro-*p*-xylene $C_8H_6Cl_4$ 

MW, 244

Needles from  $EtOH-Et_2O$ . M.p.  $222^\circ$  ( $218^\circ$ ).  
Sol.  $EtOH$ ,  $Et_2O$ ,  $C_6H_6$ .

Bureš, Rubeš, *Chem. Zentr.*, 1929, **I**, 507.Datta, Fernandes, *J. Am. Chem. Soc.*, 1916, **38**, 1811.

## Tetracosane

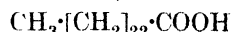
 $C_{24}H_{50}$ 

MW, 338

Isolated from paraffin from low-temperature  
coal tar. Cryst. from  $Et_2O$ . M.p.  $54^\circ$ . B.p.  
 $237-40^\circ/15$  mm. ( $243-4^\circ/15$  mm.).  $n_D^{20}$  1.43026,  
 $n_D^{25}$  1.42448.

Levene, West, *J. Biol. Chem.*, 1914, **18**, 478.Gluud, *Ber.*, 1919, **52**, 1040.Krafft, *Ber.*, 1882, **15**, 1718.

**Tetracosanic Acid** (*n-Tetracosanoic acid*.  
See also Lignoceric Acid)

 $C_{24}H_{48}O_2$ 

MW, 368.

Plates from  $AcOH$ . M.p.  $87.5-88^\circ$  ( $85-6^\circ$ ).

Anhydride:  $C_{48}H_{94}O_3$ . MW, 718. M.p.  
 $86.0-86.3^\circ$ .

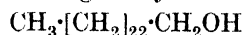
Me ester:  $C_{25}H_{50}O_2$ . MW, 382. M.p.  $59.5-60^\circ$ .

Et ester:  $C_{26}H_{52}O_2$ . MW, 396. M.p.  $56-7^\circ$ .  
B.p.  $198-9^\circ/0.24$  mm.

Nitrile:  $C_{24}H_{47}N$ . MW, 349. M.p.  $61.2^\circ$   
( $55-6^\circ$ ).

Meyer, Brod, Soyka, *Monatsh.*, 1913, **34**, 1133.Levene, West, Allen, van der Scheer, *J. Biol. Chem.*, 1915, **23**, 75.Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 905.Francis, Piper, Malkin, *Proc. Roy. Soc.*, 1930, **128 A**, 214.Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2504.Brigl, *Z. physiol. chem.*, 1915, **95**, 161.

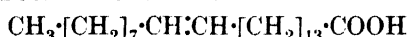
**Tetracosanol** (1-Hydroxytetracosane tetracosyl  
alcohol. See also Lignoceryl Alcohol)

 $C_{24}H_{50}O$ 

MW, 354.

M.p.  $76.5-77.5^\circ$ . B.p.  $210-210.5^\circ/0.4$  mm.Acetyl: m.p.  $57^\circ$ .Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 915.Taylor, Levene, *J. Biol. Chem.*, 1928, **80**, 609.Brigl, Fuchs, *Z. physiol. chem.*, 1922, **119**, 280.Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2504.



**14-Tetracosenic Acid**

$\text{C}_{24}\text{H}_{46}\text{O}_2$  MW, 366

*Cis* :

See Nervonic Acid.

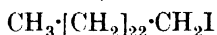
*Trans* :

Cryst. from EtOH. M.p.  $61^\circ$ .

Hale, Lycan, Adams, *J. Am. Chem. Soc.*, 1930, **52**, 4538.

**Tetracosyl Alcohol.**

See Tetracosanol.

**Tetracosyl iodide (1-Iodotetracosane)**

$\text{C}_{24}\text{H}_{49}\text{I}$  MW, 464

M.p.  $54.5\text{--}55.5^\circ$ . B.p.  $207\text{--}9^\circ/0.35$  mm.

Levene, Taylor, *J. Biol. Chem.*, 1924, **59**, 917.

Bleyberg, Ulrich, *Ber.*, 1931, **64**, 2504.

**Tetradecahydroacridine.**

See Perhydroacridine.

**Tetradecahydroanthracene.**

See Perhydroanthracene.

**Tetradecane**

$\text{C}_{14}\text{H}_{30}$  MW, 198

M.p.  $5.5^\circ$ . B.p.  $252.5^\circ$ ,  $158^\circ/50$  mm.,  $129.5^\circ/15$  mm.,  $98.5^\circ/1$  mm.  $D_4^{20}$  0.7645.

Maman, *Chem. Zentr.*, 1936, **I**, 2332.

Krafft, *Ber.*, 1882, **15**, 1700.

Sorabji, *J. Chem. Soc.*, 1885, **47**, 41.

**Tetradecane-1 : 14-dicarboxylic Acid.**

See Thapsic Acid.

**Tetradecanol-1.**

See Tetradecyl Alcohol.

**Tetradecanol-3.**

See Ethylundecylcarbinol.

**Tetradecanone-3.**

See Ethyl undecyl Ketone.

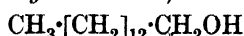
**Tetradecylacetylene (Hexadecine-1)**

$\text{C}_{16}\text{H}_{30}$  MW, 222

M.p.  $15^\circ$ . B.p.  $155^\circ/15$  mm.  $D^{20}$  0.7965.

Krafft, *Ber.*, 1896, **29**, 2236.

**Tetradecyl Alcohol (Tetradecanol-1, myristyl alcohol 1-hydroxytetradecane)**



$\text{C}_{14}\text{H}_{30}\text{O}$  MW, 214

Cryst. from EtOH.Aq. M.p.  $39\text{--}39.5^\circ$ . B.p.  $170\text{--}3^\circ/20$  mm.,  $160^\circ/10$  mm.  $D_4^{20}$  0.8236,  $D_4^{20}$  0.8153.

*Formyl* : b.p.  $166^\circ/17$  mm.

*Acetyl* : m.p.  $14^\circ$ .  $D_4^{25}$  0.8581.  $n_D^{20}$  1.4373.

*Myristyl* : plates from EtOH. M.p.  $43^\circ$ .

*p-Toluenesulphonyl* : m.p.  $35^\circ$ .

*p-Bromobenzenesulphonyl* : m.p.  $51.5^\circ$ .

*p-Methoxyphenylurethane* : m.p.  $83^\circ$ .

*3 : 4-Dimethoxyphenylurethane* : m.p.  $79.5^\circ$ .

*Nitrate* : liq. B.p.  $175\text{--}80^\circ/12$  mm. slight decomp. Spar. sol. EtOH.

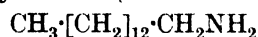
Ford, Marvel, *Organic Syntheses*, 1930, **X**, 64.

Adkins, Folkers, *J. Am. Chem. Soc.*, 1931, **53**, 1096.

Ruhoff, Reid, *J. Am. Chem. Soc.*, 1933, **55**, 3825.

**Tetradecylaldehyde.**

See Myristic Aldehyde.

**Tetradecylamine (1-Aminotetradecane)**

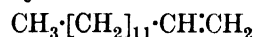
$\text{C}_{14}\text{H}_{31}\text{N}$  MW, 213

M.p.  $37^\circ$ . B.p.  $162^\circ/15$  mm.

*Acid succinate* : needles. M.p.  $123^\circ$ .

Shukoff, Schestakoff, *J. prakt. Chem.*, 1903, **67**, 419.

Krafft, *Ber.*, 1890, **23**, 2361.

**1-Tetradecylene**

$\text{C}_{14}\text{H}_{28}$  MW, 196

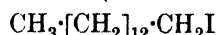
M.p.  $-12^\circ$ . B.p.  $124.5\text{--}125^\circ/15$  mm.  $D_4^{15}$  0.7745,  $D_4^{20}$  0.7683.  $n_D^{22}$  1.4392.

Klepper, *Chem. Abstracts*, 1929, **23**, 2897.

Krafft, *Ber.*, 1883, **16**, 3021.

**Tetradecylic Acid.**

See Myristic Acid.

**Tetradecyl iodide (1-Iodotetradecane)**

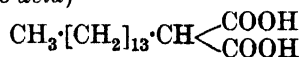
$\text{C}_{14}\text{H}_{29}\text{I}$  MW, 324

B.p.  $192\text{--}5^\circ/17.5$  mm.,  $128^\circ/0.5$  mm.

Levene, West, van der Scheer, *J. Biol. Chem.*, 1915, **20**, 529.

Majima, Nakamura, *Ber.*, 1913, **46**, 4094.

**Tetradecylmalonic Acid (Pentadecane-1 : 1-dicarboxylic acid)**



$\text{C}_{17}\text{H}_{32}\text{O}_4$  MW, 300

Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $123\text{--}4^\circ$  ( $117\text{--}18^\circ$ ). Very sol. boiling AcOH. Spar. sol. cold EtOH. Insol.  $\text{Et}_2\text{O}$ .

*Di-Et ester* :  $\text{C}_{21}\text{H}_{40}\text{O}_4$ . MW, 356. Oil. B.p.  $190^\circ/3$  mm.

**Tetraethylammonium bromide**

689

*Monoamide*:  $C_{17}H_{33}O_3N$ . MW, 299. Glistening cryst. Sol. boiling EtOH. Insol.  $H_2O$ ,  $Et_2O$ . Heat  $\rightarrow$  palmitic amide.

*Mononitrile*: 1-cyanopalmitic acid.  $C_{17}H_{31}O_2N$ . MW, 281. Leaflets from AcOH. M.p. 75–6°.

Chargaff, *Ber.*, 1932, 65, 752.

Hell, Jordanow, *Ber.*, 1891, 24, 990.

**Tetraethylammonium bromide**

$(C_2H_5)_4NBr$  MW, 210

Cryst. from EtOH. Very sol.  $H_2O$ , EtOH,  $CHCl_3$ .  $D_4^{20}$  1.3880.

$B, Br_2$ : orange-red needles from EtOH. M.p. 78°  $\rightarrow$  dark red liq.

*Add. comp. with thiourea*:  $C_8H_{20}NBr + 2CH_4N_2S$ . Prisms. M.p. 159–60°.

Wagner, *Zeitschrift für Krystallographie und Mineralogie*, 1907, 43, 190.

Walden, *Chem. Zentr.*, 1912, I, 1958.

**Tetraethylammonium chloride**

$(C_2H_5)_4NCl$  MW, 165.5

Very hygroscopic. Very sol.  $H_2O$ , EtOH,  $CHCl_3$ ,  $Me_2CO$ .  $D_4^{25}$  1.1115.

$B, 4H_2O$ : prisms. M.p. 37.5°

See last reference above and also

Wagner, *Zeitschrift für Krystallographie und Mineralogie*, 1907, 43, 189.

**Tetraethylammonium hydroxide**

$(C_2H_5)_4N \cdot OH$  MW, 147

Known only in solution and in form of hydrates. Aq. sol. reacts alkaline and has bitter taste. Sucrose +  $CuSO_4 \rightarrow$  blue sol. Hydrolyses fats.

$B, 4H_2O$ : needles. M.p. 49–50°. Very sol.  $H_2O$ .

$B, 6H_2O$ : m.p. 55°.

Hofmann, *Ann.*, 1851, 78, 263.

Crichton, *J. Chem. Soc.*, 1907, 91, 1794.

**Tetraethylammonium iodide**

$(C_2H_5)_4NI$  MW, 257

Cryst. from warm  $H_2O$ . Does not melt below 200°. Sol.  $H_2O$ . Mod. sol.  $CHCl_3$ . Insol.  $Et_2O$ .  $D_4^{20}$  1.559.

$B, I_2$ : prisms. M.p. 143°. Sol.  $Me_2CO$ , hot EtOH. Insol.  $Et_2O$ .

$B, I_6$ : cryst. M.p. 108°.

Dict. of Org. Comp.—III.

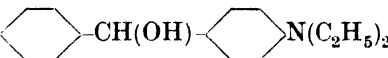
**4 : 4'-Tetraethyldiaminotriphenylmethane**

*Add. comp. with thiourea*:  $C_8H_{20}NI + 2CH_4N_2S$ . M.p. 135°.

Walden, *Chem. Zentr.*, 1914, I, 603.

Wagner, *Zeitschrift für Krystallographie und Mineralogie*, 1907, 43, 191.

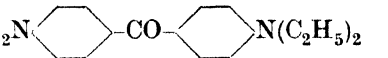
**4 : 4'-Tetraethyldiaminobenzhydrol**

$(C_2H_5)_2N$    $N(C_2H_5)_2$  MW, 326

Cryst. from pet. ether. M.p. 78°.

Votoček, Köhler, *Ber.*, 1913, 46, 1761.

**4 : 4'-Tetraethyldiaminobenzophenone**

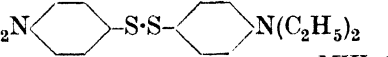
$(C_2H_5)_2N$    $N(C_2H_5)_2$  MW, 324

Leaflets from EtOH. M.p. 95–6°. Intermediate for triphenylmethane dyes.

*Anil*: m.p. 124–5°.

Votoček, Köhler, *Ber.*, 1913, 46, 1761.

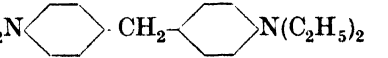
**4 : 4'-Tetraethyldiaminodiphenyl disulphide (Dithiodiethylaniline, NN'-tetra-ethylthioaniline)**

$(C_2H_5)_2N$    $N(C_2H_5)_2$  MW, 360

Prisms from EtOH. M.p. 72°. Sol.  $CS_2$ . Spar. sol. EtOH,  $C_6H_6$ , ligroin, hot  $Et_2O$ .

Holzmann, *Ber.*, 1887, 20, 1637.

**4 : 4'-Tetraethyldiaminodiphenylmethane**

$(C_2H_5)_2N$    $N(C_2H_5)_2$  MW, 310

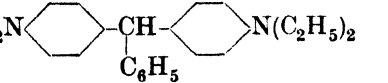
Cryst. from EtOH. M.p. 41°. B.p. 253°/10 mm.

*Picrate*: leaflets. M.p. 191°.

Votoček, Köhler, *Ber.*, 1913, 46, 1760.

v. Braun, Kruber, *Ber.*, 1912, 45, 2996.

**4 : 4'-Tetraethyldiaminotriphenylmethane**

$(C_2H_5)_2N$    $N(C_2H_5)_2$  MW, 386

Needles from EtOH. M.p. 62°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Very spar. sol.  $H_2O$ .

*Chloroplatinate*: orange-red needles. M.p. 254-7°.

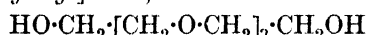
Doebner, *Ann.*, 1883, **217**, 265.

Decker, Becker, *Ann.*, 1913, **395**, 372.

**N : N'-Tetraethyldithioaniline.**

See Tetraethyldiaminodiphenyl disulphide.

**Tetraethylene Glycol** (*Diethyl ether*  $\beta$ -hydroxyethyl] ether)



$\text{C}_8\text{H}_{18}\text{O}_5$  MW, 194

B.p. 328°, 198°/14 mm., 157°/2 mm.  $D_4^{15}$  1.1285.  $n_D^{20}$  1.4609.

*Cyclic carbonate*: m.p. 42-4°. B.p. 128-30°/1 mm.  $D_4^{20}$  1.1961.  $n_D^{50}$  1.4569. Has slight odour of musk. Employed as perfume.

du Pont, F.P., 768,807, (*Chem. Abstracts*, 1935, **29**, 557).

Matignon, Moureu, Dodé, *Bull. soc. chim.*, 1934, **1**, 1308.

Hill, Carothers, *J. Am. Chem. Soc.*, 1933, **55**, 5034.

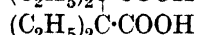
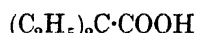
**Tetraethylnaphthylenediamine.**

See under Naphthylenediamine.

**Tetraethylphenylenediamine.**

See under Phenylenediamine.

**Tetraethylsuccinic Acid**



$\text{C}_{12}\text{H}_{22}\text{O}_4$  MW, 230

Cryst. from  $\text{Et}_2\text{O}$ -pet. ether. M.p. 149°  $\rightarrow$  anhydride. Very sol.  $\text{Et}_2\text{O}$ . Sol.  $\text{EtOH}$ . Spar. sol.  $\text{H}_2\text{O}$ , pet. ether.  $k = 4.4 \times 10^{-4}$  at 25°.

*Di-Et ester*:  $\text{C}_{16}\text{H}_{30}\text{O}_4$ . MW, 286. B.p. 168-72°/25 mm.  $D_4^{20}$  1.011.

*Diamide*:  $\text{C}_{12}\text{H}_{24}\text{O}_2\text{N}_2$ . MW, 228. Cryst. from pet. ether. M.p. 49-50°. B.p. 276-7°.

*Dinitrile*:  $\text{C}_{12}\text{H}_{20}\text{N}_2$ . MW, 192. Plates. M.p. 100-1°.

*Anhydride*:  $\text{C}_{12}\text{H}_{20}\text{O}_3$ . MW, 212. Prisms from pet. ether, with odour resembling camphor. M.p. 86°. B.p. 270°.

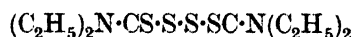
Verkade, Hartmann, *Rec. trav. chim.*, 1933, **52**, 945.

Walker, Walker, *J. Chem. Soc.*, 1905, **87**, 964.

**Tetraethylthiourea.**

See under Thiourea.

**Tetraethylthiuram disulphide**



$\text{C}_{10}\text{H}_{20}\text{N}_2\text{S}_4$  MW, 296

Cryst. M.p. 70°. Sol. warm  $\text{EtOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ . KOH fusion  $\rightarrow$  diethylamine. Rubber vulcanisation accelerator.

Cummings, Simmons, *Ind. Eng. Chem.*, 1928, **20**, 1173.

Naugatuck, U.S.P., 1,782,111, (*Chem. Abstracts*, 1931, **25**, 303).

Roessler and Hasslacher, U.S.P., 1,796,977, (*Chem. Abstracts*, 1931, **25**, 2598).

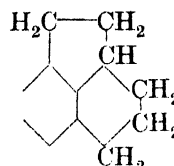
**Tetraethylurea.**

See under Urea.

**Tetrafluoromethane.**

See Carbon tetrafluoride.

**Tetrahydroacenaphthene** (*Tetraphthene*)



$\text{C}_{12}\text{H}_{14}$

MW, 158

M.p. 12°. B.p. 249.5°/719 mm., 115°/12 mm. Turns slightly yellow in air.  $D_4^{21}$  1.0290.  $n_D$  1.5777. Decolourises  $\text{KMnO}_4$ . Reacts very vigorously with  $\text{CrO}_3 + \text{H}_2\text{SO}_4$ .

*Picrate*: m.p. about 152-3°.

Padoa, Fabris, *Gazz. chim. ital.*, 1909, **39**, i, 331.

Bayer, D.R.P., 306,724, (*Chem. Zentr.*, 1918, II, 420).

v. Braun, Kirschbaum, *Ber.*, 1922, **55**, 1682.

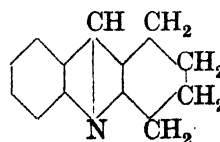
Goswami, *Compt. rend.*, 1924, **179**, 1269.

Fleischer, Siefert, *Ann.*, 1921, **422**, 304.

**$\Delta^1$ -Tetrahydroacetophenone.**

See 1-Acetocyclohexene.

**1 : 2 : 3 : 4-Tetrahydroacridine**



$\text{C}_{13}\text{H}_{13}\text{N}$

MW, 183

Cryst. from pet. ether. M.p. 54.5°.

$B\cdot HNO_3$ : pale yellow plates from  $\text{EtOH}$ . M.p. 160°.

$B_2\cdot H_2SO_4$ : needles from  $\text{EtOH}$ . M.p. 220°.

$B_2\cdot H_2PtCl_6$ : brownish-yellow needles from alc.  $\text{HCl}$ . M.p. 233-5°.

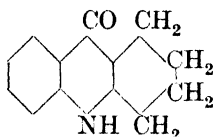
*Picrate*: yellow needles. M.p. 208° decomp.

*Methiodide* : needles from EtOH-Et<sub>2</sub>O. M.p. 202-4°.

Perkin, Sedgwick, *J. Chem. Soc.*, 1924, 125, 2446.

Borsche, *Ber.*, 1908, 41, 2206.

## 1 : 2 : 3 : 4-Tetrahydroacridone

C<sub>13</sub>H<sub>13</sub>ON

MW, 199

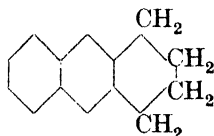
Needles from EtOH or aniline. M.p. 358°. B.p. 360-70°/10 mm. Very sol. boiling aniline. Sol. EtOH with green fluor. Sublimation in air, or heat with H<sub>2</sub>SO<sub>4</sub> → acridone.

Tiedtke, *Ber.*, 1909, 42, 624.

Perkin, Sedgwick, *J. Chem. Soc.*, 1924, 125, 2441.

v. Braun, Heymons, Manz, *Ber.*, 1931, 64, 233.

Riedel, de Haen A.G., D.R.P., 532,397, (*Chem. Abstracts*, 1932, 26, 151).

1 : 2 : 3 : 4-Tetrahydroanthracene (*Tetrahcene*)C<sub>14</sub>H<sub>14</sub>

MW, 182

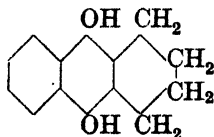
Leaflets from EtOH. M.p. 103-5°. B.p. 170-3°/14 mm. CrO<sub>3</sub> in AcOH → 1 : 2 : 3 : 4-tetrahydroanthraquinone.

*Picrate* : reddish-yellow needles from EtOH. M.p. 116-17°.

Schroeter, *Ber.*, 1924, 57, 2013; D.R.P., 463,830, (*Chem. Abstracts*, 1928, 22, 4134).

Klepper, *Chem. Abstracts*, 1929, 23, 3897. Prokopetz, Khadzhinov, *Chem. Abstracts*, 1935, 29, 7319.

## 1 : 2 : 3 : 4-Tetrahydroanthrahydroquinone (9 : 10-Dihydroxy-1 : 2 : 3 : 4-tetrahydroanthracene)

C<sub>14</sub>H<sub>14</sub>O<sub>2</sub>

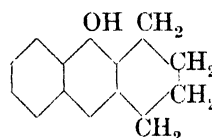
MW, 214

Greenish needles from AcOH. M.p. 208-16° decomp. Sol. alkalis with yellow col. Conc. H<sub>2</sub>SO<sub>4</sub> → reddish-brown sol.

*Diacetyl* : cryst. from AcOH or EtOH. M.p. 204-6°.

Skita, *Ber.*, 1925, 58, 2694.

## 1 : 2 : 3 : 4-Tetrahydroanthranol (9-Hydroxy-1 : 2 : 3 : 4-tetrahydroanthracene)

C<sub>14</sub>H<sub>14</sub>O

MW, 198

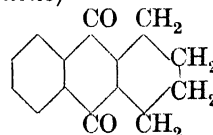
Pale yellow cryst. from ligroin. M.p. 108°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Very spar. sol. pet. ether. Sol. alkalis with strong yellowish-green fluor. No col. with FeCl<sub>3</sub>.

*Me ether* : C<sub>15</sub>H<sub>16</sub>O. MW, 212. Reddish-yellow oil. B.p. 197°/14 mm., slight decomp.

*Acetyl* : needles from EtOH. M.p. 109°. Spar. sol. EtOH with violet fluor.

*Benzoyl* : m.p. 142°.

v. Braun, Bayer, *Ber.*, 1925, 58, 2675.

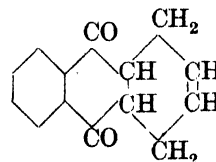
1 : 2 : 3 : 4-Tetrahydroanthraquinone (*Tetrahcenequinone*)C<sub>14</sub>H<sub>12</sub>O<sub>2</sub>

MW, 212

Golden-yellow needles from AcOH or AcOEt. M.p. 156-8°.

Schroeter, *Ber.*, 1924, 57, 2014; D.R.P., 463,830, (*Chem. Abstracts*, 1928, 22, 4134).

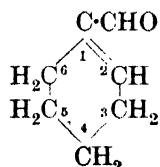
Skita, *Ber.*, 1925, 58, 2694.

 $\Delta^2$ -TetrahydroanthraquinoneC<sub>14</sub>H<sub>12</sub>O<sub>2</sub>

MW, 212

Cryst. from EtOH or ligroin. M.p. 102-3°. Sol. usual solvents on warming.

Diels, Alder, *Ann.*, 1928, 460, 110.

$\Delta^1$ -Tetrahydrobenzaldehyde (*Cyclohexene-1-aldehyde*) $C_7H_{10}O$ 

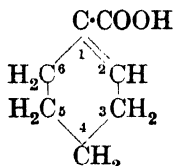
MW, 110

Oil.

*Oxime*: cryst. from ligroin. M.p. 97–9°.*Semicarbazone*: cryst. M.p. 212–13°.Borsche, Schmidt, *Ber.*, 1910, **43**, 3400.v. Braun, Danziger, *Ber.*, 1913, **46**, 107.Wallach, *Ann.*, 1908, **359**, 292. $\Delta^3$ -Tetrahydrobenzaldehyde (*Cyclohexene-3-aldehyde*).Oil. B.p. 163.5–164.5°, 58°/17 mm., 51–2°/13 mm.  $D_4^{20}$  0.9524. Polymerises very readily.*Semicarbazone*: cryst. from  $C_6H_6$ -ligroin or MeOH.Aq. M.p. 153.5–154.5°.Sobecki, *Ber.*, 1910, **43**, 1040.Diels, Alder, *Ann.*, 1928, **460**, 121.

## Tetrahydrobenzene.

See Cyclohexene.

 $\Delta^1$ -Tetrahydrobenzoic Acid (*Cyclohexene-1-carboxylic acid*) $C_7H_{10}O_2$ 

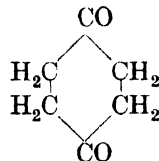
MW, 126

Plates. M.p. 38°. B.p. 238–40°/683 mm., 138°/14 mm., 133–4°/11 mm., 107°/3 mm.  $D_4^{17.2}$  1.0717.  $n_D^{20}$  1.49023.*Me ester*:  $C_8H_{12}O_2$ . MW, 140. B.p. 193.5–194.5°.  $D_4^{20}$  1.05607,  $D_D^{20}$  1.04364.*Et ester*:  $C_9H_{14}O_2$ . MW, 154. B.p. 206–8°, 143°/100 mm., 96°/16 mm., 84–6°/12 mm.  $D_4^{14}$  1.0032.  $n_D^{20}$  1.47167.*l-Menthyl ester*: b.p. 178°/12 mm.  $[\alpha]_D^{20}$  –74.64° in EtOH.*Chloride*:  $C_7H_9OCl$ . MW, 144.5. B.p. 86°/11 mm.*Nitrile*:  $C_7H_9N$ . MW, 107. B.p. 81°/12 mm.*Amide*:  $C_7H_{11}ON$ . MW, 125. Prisms from EtOH.Aq. M.p. 127–8°.Darzens, Rost, *Compt. rend.*, 1911, **153**, 773.Auwers, Krollpfeiffer, *Ber.*, 1915, **48**, 1396.Ruzicka, Brugger, *Helv. Chim. Acta*, 1926, **9**, 402.Boorman, Linstead, *J. Chem. Soc.*, 1935, 261. $\Delta^2$ -Tetrahydrobenzoic Acid (*Cyclohexene-3-carboxylic acid*).Liq. B.p. 234–5°, 130°/18 mm.  $D_4^{20}$  1.0820.  $n_D^{20}$  1.4814. 1.34 parts sol. 100 parts  $H_2O$  at 20°.  $k = 3.05 \times 10^{-5}$  at 25°. Oxidises slowly in air to benzoic acid.*Me ester*: b.p. 188–9°.  $D_4^{20}$  1.0433.*l-Menthyl ester*: b.p. 176°/12 mm.  $[\alpha]_D^{20}$  –59.44° in EtOH.*Amide*: leaflets or prisms from EtOH. M.p. 144°.

See last reference above and also

Aschan, *Ann.*, 1892, **271**, 234. $\Delta^3$ -Tetrahydrobenzoic Acid (*Cyclohexene-4-carboxylic acid*).

M.p. about 13°. B.p. 237°/748 mm. Oxidises in air only very slowly.

Perkin, Tattersall, *J. Chem. Soc.*, 1907, **91**, 490.Sobecki, *Ber.*, 1910, **43**, 1039.Tetrahydro-*p*-benzoquinone (*Cyclohexan-dione-1 : 4, 1 : 4-diketocyclohexane*) $C_6H_8O_2$ 

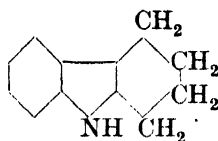
MW, 112

Needles from pet. ether, plates from  $H_2O$ . M.p. 78°. Sol. usual solvents. Warm  $FeCl_3 \rightarrow p$ -benzoquinone.*Di-Me acetal*: m.p. 80–1°.*Di-Et acetal*: plates from EtOH. M.p. 89°.*Dioxime*: cryst. from  $H_2O$ . M.p. 188°. Mod. sol.  $H_2O$ .*Monosemicarbazone*: cryst. from EtOH. M.p. 221–2° decomp. Mod. sol. EtOH.*Di-thiosemicarbazone*: cryst. M.p. 210–15°.Meerwein, *Ann.*, 1913, **398**, 248.Baeyer, *Ann.*, 1894, **278**, 91.Stollé, *Ber.*, 1901, **34**, 1344.Piloty, Steinbock, *Ber.*, 1902, **35**, 3109.

## Tetrahydroberberine.

See Canadine.

## Tetrahydrocarbazole

 $C_{12}H_{13}N$ 

MW, 171

Leaflets from EtOH.Aq. M.p. 120° (116°). B.p. 325–30°. Turns brown in air. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. Volatile in superheated steam. KOH fusion → indole-2-carboxylic acid.

N-Acetyl: prisms from EtOH. M.p. 77°.

N-Benzoyl: m.p. 85°.

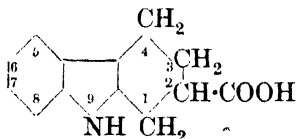
Picrate: dark red leaflets. M.p. 147°.

Bucherer, Brandt, *J. prakt. Chem.*, 1934, 140, 129.

Perkin, Plant, *J. Chem. Soc.*, 1921, 119, 1825.

Chem. Fabr. Weiler ter Meer, D.R.P., 374,098, (*Chem. Abstracts*, 1924, 18, 2175).

## Tetrahydrocarbazole-2-(or 4-)carboxylic Acid

 $C_{13}H_{13}O_2N$ 

MW, 215

Cryst. M.p. 230°.

Baeyer, Tutein, *Ber.*, 1889, 22, 2185.

## Tetrahydrocarbazole-3-carboxylic Acid.

Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 195°. Sol. EtOH. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether.

Perkin, *J. Chem. Soc.*, 1904, 85, 428.

## Tetrahydrocarbazole-5-(or 7-)carboxylic Acid.

Prisms from AcOH. M.p. 287°.

Me ester: needles from MeOH. M.p. 155°.

Et ester: plates from EtOH.Aq. M.p. 146°.

See previous reference.

## Tetrahydrocarbazole-6-carboxylic Acid.

Plates from EtOH.Aq. M.p. 282°.

Me ester: leaflets from AcOH.Aq. M.p. 158°.

Et ester: prisms from EtOH.Aq. M.p. 119°.

See previous reference.

## Tetrahydrocarbazole-7-(or 5-)carboxylic Acid.

Prisms from AcOH. M.p. 210°.

Me ester: prisms from EtOH.Aq. M.p. 93°.

See previous reference.

## Tetrahydrocarbazole-8-carboxylic Acid.

Prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 203°.

Me ester: C<sub>14</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 229. Prisms from MeOH. M.p. 124°.

Et ester: C<sub>15</sub>H<sub>17</sub>O<sub>2</sub>N. MW, 243. Needles from EtOH.Aq. M.p. 76°.

Collar, Plant, *J. Chem. Soc.*, 1926, 809.

## Tetrahydrocarbazole-9-carboxylic Acid.

Not isolated as pure comp. owing to ease with which it loses CO<sub>2</sub>.

Et ester: needles from EtOH. M.p. 65°.

Perkin, Plant, *J. Chem. Soc.*, 1923, 123, 691.

## Tetrahydrocarveol.

See Carvomenthol.

## Tetrahydrocarvone.

See p-Menthanone-2.

## Tetrahydrocinchoninic Acid.

See Tetrahydroquinoline-4-carboxylic Acid.

## 1 : 2 : 3 : 6-Tetrahydro-ψ-cumene.

See 1 : 4 : 5-Trimethylcyclohexene.

## Tetrahydrocymene.

See Menthene.

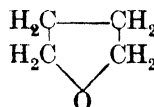
## Tetrahydroeucarvone.

See 2 : 6 : 6-Trimethylcycloheptanone.

## Tetrahydroferulene.

See under Ferulene.

## Tetrahydrofuran (Tetramethylene oxide)

 $C_4H_8O$ 

MW, 72

Oil. F.p. – 65°. B.p. 64–5°. Sol. most org. solvents. Mod. sol. H<sub>2</sub>O.  $D_4^{21}$  0.888.  $n_D^{21}$  1.40762. Bitter taste.

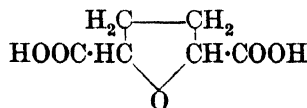
Starr, Hixon, *Organic Syntheses*, 1936, XVI, 77.

Bourguignon, *Chem. Zentr.*, 1908, I, 1630.

## Tetrahydrofuran-carboxylic Acid.

See Tetrahydropyromucic Acid.

## Tetrahydrofuran-2 : 5-dicarboxylic Acid

 $C_6H_8O_5$ 

MW, 160

Cis :

Cryst. from AcOEt–pet. ether. M.p. 124–5°. Very sol. H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO, AcOH. Mod. sol. Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether.

*Dianilide*: plates from EtOH. M.p. 208–9° decomp. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>. Spar. sol. EtOH, hot C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*Anhydride*: needles from CHCl<sub>3</sub>-pet. ether. M.p. 128–9°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOEt. Insol. pet. ether. Sublimes readily.

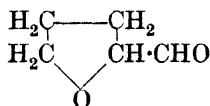
*Trans*:

Cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. 59–61°, anhyd. 94–5°. Very sol. H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO, AcOH. Mod. sol. boiling Et<sub>2</sub>O. Spar. sol. boiling C<sub>6</sub>H<sub>6</sub>, toluene, pet. ether.

Lean, *J. Chem. Soc.*, 1900, **77**, 110.

Le Sueur, Haas, *J. Chem. Soc.*, 1910, **97**, 181.

**Tetrahydrofurfural** (*Tetrahydro-2-furoic aldehyde*)



C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>

MW, 100

Mobile oil. B.p. 142–3°/779 mm., 45–7°/29 mm. D<sub>4</sub><sup>20</sup> 1.0727. n<sub>D</sub><sup>20</sup> 1.43658. Reduces hot Fehling's instantly, NH<sub>3</sub>.AgNO<sub>3</sub> on standing. Decolourises alk. KMnO<sub>4</sub>. Conc. HCl → intense brick-red col.

*Di-Et acetal*: b.p. 194.5° (187–90°).

*Di-[β-ethoxyethyl]-acetal*: b.p. 131–6°/4–5 mm.

*Diacetate*: b.p. 134–6°/16 mm. (133°/29 mm.). D<sub>4</sub><sup>20</sup> 1.1495. n<sub>D</sub><sup>20</sup> 1.44052. Sol. usual org. solvents. Spar. sol. H<sub>2</sub>O.

*Phenylbenzylhydrazone*: pale yellow needles from MeOH. M.p. 67°.

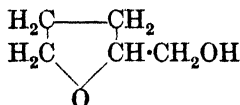
Scheibler, Sotscheck, Friese, *Ber.*, 1925, **58**, 1961; 1924, **57**, 1443.

Burdick, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 440.

Covert, Connor, Adkins, *J. Am. Chem. Soc.*, 1932, **54**, 1656.

Minné, Adkins, *J. Am. Chem. Soc.*, 1933, **55**, 304.

**Tetrahydrofurfuryl Alcohol**



C<sub>5</sub>H<sub>10</sub>O<sub>2</sub>

MW, 102

B.p. 177–8°/750 mm., 80–2°/20 mm. D<sub>4</sub><sup>25</sup> 1.1326. n<sub>D</sub><sup>25</sup> 1.4505. Absorbs moisture from the air. Solvent for fats, waxes, resins, etc.

*Et ether*: C<sub>7</sub>H<sub>14</sub>O<sub>2</sub>. MW, 130. B.p. 152–4°/726 mm., 47–55°/11 mm.

*Acetyl*: b.p. 192–4°/740 mm., 88–90°/18 mm. D<sub>4</sub><sup>25</sup> 1.0624. n<sub>D</sub><sup>25</sup> 1.4350.

*Iodoacetyl*: b.p. 130°/5 mm.

*Propionyl*: b.p. 204–7°/756 mm., 85–7°/3 mm. D<sub>4</sub><sup>20</sup> 1.044.

*Butyryl*: b.p. 225–7°/759 mm., 102–4°/4 mm. D<sub>20</sub> 1.012.

*Valeryl*: b.p. 238–40°/756 mm., 97–9°/2 mm. D<sub>20</sub> 0.999.

*Pyruryl*: b.p. 110–30°/17 mm. *Semicarbazone*: leaflets. M.p. 184–6°.

*Benzoyl*: b.p. 300–2°/750 mm., 138–40°/2 mm. D<sub>20</sub> 1.137.

*Phenylurethane*: cryst. from pet. ether. M.p. 61°.

*Diphenylurethane*: plates from MeOH. M.p. 81°.

Wienhaus, *Ber.*, 1920, **53**, 1656.

Zanetti, *J. Am. Chem. Soc.*, 1928, **50**, 1821.

du Pont, E.P., 337,296, (*Chem. Abstracts*, 1931, **25**, 1844).

Böhme, E.P., 388,703, (*Chem. Abstracts*, 1933, **27**, 4547).

Hewlett, *Chem. Abstracts*, 1933, **27**, 980.

Burdick, Adkins, *J. Am. Chem. Soc.*, 1934, **56**, 441.

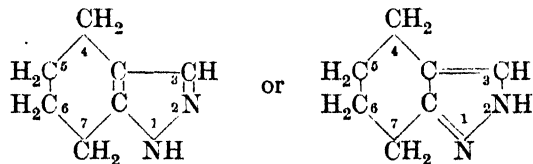
**Tetrahydrofuroic Acid.**

See Tetrahydropyromucic Acid.

**Tetrahydroiminazolone-2.**

See Ethyleneurea.

**4 : 5 : 6 : 7-Tetrahydroindazole**



C<sub>7</sub>H<sub>10</sub>N<sub>2</sub>

MW, 122

Cryst. M.p. 84°.

*1-Benzoyl*: prisms from pet. ether. M.p. 58.5–60°. B.p. 208°/13 mm. Sol. most org. solvents.

*1-o-Nitrobenzoyl*: cryst. from EtOH. M.p. 148–9°.

*1-m-Nitrobenzoyl*: needles from EtOH. M.p. 140–2°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, Et<sub>2</sub>O.

*2-o-Nitrobenzoyl*: cryst. from EtOH. M.p. 179–80°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Less sol. EtOH, Et<sub>2</sub>O.

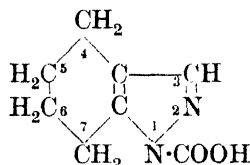
**4 : 5 : 6 : 7-Tetrahydroindazole-1-carboxylic Acid** 695

2-m-Nitrobenzoyl : leaflets from EtOH. M.p. 118-118.5°.

Auwers, *Ann.*, 1927, **453**, 227.

Wallach, *Ann.*, 1903, **329**, 118.

**4 : 5 : 6 : 7-Tetrahydroindazole-1-carboxylic Acid**



$C_8H_{10}O_2N_2$  MW, 166

*Et ester* :  $C_{10}H_{14}O_2N_2$ . MW, 194. B.p. 170°/18 mm.  $D_4^{20}$  1.127.  $n_D^{20}$  1.5147.

*Chloride* :  $C_8H_9ON_2Cl$ . MW, 184.5. Cryst. powder from  $C_6H_6$ . M.p. 162-5°.

*Amide* : cryst. from AcOH.Aq. M.p. 187°.

Auwers, *Ann.*, 1927, **453**, 230.

**4 : 5 : 6 : 7-Tetrahydroindazole-2-carboxylic Acid.**

*Et ester* : b.p. 170°/18 mm.  $D_4^{20}$  1.138.  $n_D^{20}$  1.5120.

*Amide* : cryst. M.p. 186-8°.

See previous reference and also

Wallach, *Ann.*, 1903, **329**, 117.

**4 : 5 : 6 : 7-Tetrahydroindazole-3-carboxylic Acid.**

Needles from AcOH. M.p. 254°. Mod. sol. AcOH. Spar. sol. EtOH. Very spar. sol.  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ .

*Me ester* : 1-o-nitrobenzoyl, needles from AcOH. M.p. 193-4°. Sol. hot EtOH, AcOH. Spar. sol.  $C_6H_6$ .

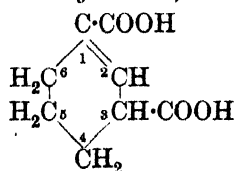
*Et ester* : cryst. from pet. ether. M.p. 106-7°. Sol. most org. solvents.

1-o-Nitrobenzoyl : m.p. 148-9°.

Auwers, *Ann.*, 1927, **453**, 232.

Auwers, Ernecke, Conrad, Ottens, *Ann.*, 1929, **469**, 68.

**$\Delta^1$ -Tetrahydroisophthalic Acid (Cyclohexene-1 : 3-dicarboxylic acid)**



$C_8H_{10}O_4$  MW, 170

Cryst. from  $H_2O$ . M.p. 197-8° (168°). Very sol.  $H_2O$ , EtOH, AcOH. Spar. sol.  $CHCl_3$ ,  $C_6H_6$ , pet. ether.  $KMnO_4 \rightarrow$  succinic acid.

**1 : 2 : 3 : 4-Tetrahydroisoquinoline**

Hot KOH or HCl  $\rightarrow$   $\Delta^3$ -tetrahydroisophthalic acid.

*Mono-Me ester* :  $C_9H_{12}O_4$ . MW, 184. Exists in two forms. (i) Prisms from hexane. M.p. 59°. (ii) Oil. B.p. 172-4°/1 mm.

*Di-Me ester* :  $C_{10}H_{14}O_4$ . MW, 198. B.p. 134-5°/7 mm.

*Mono-Et ester* :  $C_{10}H_{14}O_4$ . MW, 198. Exists in two forms. (i) Prisms from pet. ether. M.p. 44-5°. (ii) Cryst. from pet. ether. M.p. 40-1°. B.p. 169-73°/1 mm.

*Di-Et ester* :  $C_{12}H_{18}O_4$ . MW, 226. B.p. 150°/12 mm.  $D_4^{20}$  1.0772.  $n_D$  1.4722.

*Amide* : prisms from  $H_2O$ . M.p. 239°.

*Anhydride* : plates from  $C_6H_6$ -pet. ether. M.p. 78-80°. Sol. warm  $C_6H_6$ . Spar. sol.  $Et_2O$ .

*Monoanilide* : needles from AcOH.Aq. M.p. 190-2°.

Kon, Nandi, *J. Chem. Soc.*, 1933, 1631.

Farmer, Richardson, *J. Chem. Soc.*, 1926, 2174.

Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 301.

**$\Delta^3$ -Tetrahydroisophthalic Acid (Cyclohexene-1 : 5-dicarboxylic acid).**

Cryst. from  $H_2O$ . M.p. 243-4°. Sol. boiling formic acid. Spar. sol. boiling  $H_2O$ .  $KMnO_4$  or  $HNO_3 \rightarrow$  isophthalic acid.

*Di-Me ester* : b.p. 140-1°/7 mm.

Farmer, Richardson, *J. Chem. Soc.*, 1926, 2176.

Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 306.

**$\Delta^4$ -Tetrahydroisophthalic Acid (Cyclohexene-3 : 5-dicarboxylic acid).**

*Cis* :

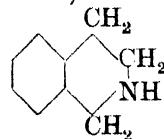
Needles from  $H_2O$ . M.p. 165°. Very sol. hot  $H_2O$ . Hot KOH  $\rightarrow$   $\Delta^3$ -tetrahydroisophthalic acid.

*Trans* :

Cryst. M.p. 225-7°. Spar. sol.  $H_2O$ .

Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 310.

**1 : 2 : 3 : 4-Tetrahydroisoquinoline (Py-Tetrahydroisoquinoline)**



$C_9H_{11}N$  MW, 133

B.p. 232-3°.  $D_4^{23-1}$  1.0642.  $n_D^{23-1}$  1.5798. Spar. sol.  $H_2O$ . Heat of comb.  $C_e$  1214 Cal. Reduces  $NH_3$ .  $AgNO_3$ .



**5 : 6 : 7 : 8-Tetrahydroisoquinoline**

*B.HCl*: plates. M.p. 195–7°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: reddish-yellow plates. M.p. 231–2°.

*Picrate*: yellow needles from EtOH. M.p. 195°.

*Picrolonate*: m.p. 260° decomp.

*N-Me*: see Isokairiline.

*N-Et*: C<sub>11</sub>H<sub>15</sub>N. MW, 161. Pale yellow oil. B.p. 225–7°. *B.HI*: pale yellow needles from H<sub>2</sub>O. M.p. 170°. *Picrate*: yellow needles from EtOH. M.p. 121°.

*N-Propyl*: C<sub>12</sub>H<sub>17</sub>N. MW, 175. B.p. 259–60°/743 mm.

*N-Isopropyl*: b.p. 256–8°/735 mm.

*N-Butyl*: C<sub>13</sub>H<sub>19</sub>N. MW, 189. B.p. 272–3°.

*N-Isoamyl*: C<sub>14</sub>H<sub>21</sub>N. MW, 203. B.p. 276–80°.

*N-Benzyl*: pale yellow oil. B.p. 194–7°/18 mm.

*N-Phenacyl*: yellow needles from EtOH. M.p. 100–1°.

*N-Acetyl*: cryst. from ligroin. M.p. 46°. B.p. 220–5°/70 mm. Sol. usual org. solvents.

*N-Benzoyl*: m.p. 129°. B.p. 245–50°/50 mm.

*N-o-Nitrobenzoyl*: needles from EtOH. M.p. 75–6°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether.

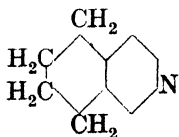
Kondo, Ochiai, *Chem. Abstracts*, 1923, 17, 3032.

Wedekind, Ney, *Ber.*, 1909, 42, 2140; 1912, 45, 1308.

Bamberger, Dieckmann, *Ber.*, 1893, 26, 1213.

Pictet, Spengler, *Ber.*, 1911, 44, 2034.

Pictet, D.R.P., 241,425, (*Chem. Zentr.*, 1912, I, 177).

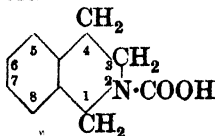
**5 : 6 : 7 : 8-Tetrahydroisoquinoline (Bz-Tetrahydroisoquinoline)**

C<sub>9</sub>H<sub>11</sub>N

MW, 133

B.p. 218°. *D*<sub>4</sub><sup>10</sup> 1.0504. *n*<sub>D</sub><sup>10</sup> 1.57263.

Yamaguchi, *Chem. Abstracts*, 1927, 21, 2696.

**1 : 2 : 3 : 4 - Tetrahydroisoquinoline - 2 - carboxylic Acid**

C<sub>10</sub>H<sub>11</sub>O<sub>2</sub>N

MW, 177

**696 1 : 2 : 3 : 4-Tetrahydronaphthalene-1 : 2-dicarboxylic Acid**

*Amide*: C<sub>10</sub>H<sub>12</sub>ON<sub>2</sub>. MW, 176. Leaflets from H<sub>2</sub>O. M.p. 169°. Spar. sol. Et<sub>2</sub>O.

*Anilide*: needles from EtOH. M.p. 144°. Spar. sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Bamberger, Dieckmann, *Ber.*, 1893, 26, 1212.

**1 : 2 : 3 : 4 - Tetrahydroisoquinoline - 3 - carboxylic Acid.**

Cryst. from EtOH.Aq. M.p. 311° decomp. Mod. sol. warm H<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>. Insol. EtOH, Et<sub>2</sub>O. Heat above m.p. → 1 : 2 : 3 : 4-tetrahydroisoquinoline.

Pictet, Spengler, *Ber.*, 1911, 44, 2034.

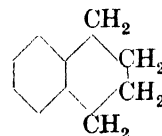
Pictet, D.R.P., 241,425, (*Chem. Zentr.*, 1912, I, 177).

**Tetrahydrolepidine.**

See 4-Methyltetrahydroquinoline.

**Tetrahydromesitylene.**

See 1 : 3 : 5-Trimethylcyclohexene.

**Tetrahydronaphthalene (Tetralin)**

C<sub>10</sub>H<sub>12</sub>

MW, 132.

M.p. – 35°. B.p. 207.3°, 90.8–91.2°/17 mm. *D*<sub>4</sub><sup>17</sup> 0.9738. *n*<sub>D</sub><sup>17</sup> 1.54529. Sp. heat 0.403 at 18°.

Heat of vap. 79.32 cal. per gram. Heat of comb. C<sub>p</sub> 1353 Cal. Crit. temp. 789°. Used extensively as solvent for org. substances. Non-toxic.

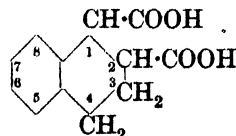
Graebe, Guye, *Ber.*, 1883, 16, 3028.

Heaton, *Journal of Oil and Colour Chemists Association*, 1923, 6, 93.

Inoue, *Chem. Abstracts*, 1924, 18, 2697.

Gewerkschaft Mathias Stinnes, D.R.P., 610,829, (*Chem. Abstracts*, 1935, 29, 5866).

I.C.I., E.P., 401,724, (*Chem. Abstracts*, 1934, 28, 2723).

**1 : 2 : 3 : 4-Tetrahydronaphthalene-1 : 2-dicarboxylic Acid**

C<sub>12</sub>H<sub>12</sub>O<sub>4</sub>

MW, 220

Needles. M.p. 193° (rapid heat.). Sol. Me<sub>2</sub>CO. Mod. sol. EtOH, AcOH, hot H<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>.

**1 : 2 : 3 : 4-Tetrahydronaphthalene-1 : 8- dicarboxylic Acid** 697 **5 : 6 : 7 : 8-Tetrahydro-2-naphthoic Acid**

*Anhydride*:  $C_{12}H_{10}O_3$ . MW, 202. Needles from MeOH. M.p. 66–7°. Sol. AcOH. Spar. sol.  $C_6H_6$ .

Auwers, Möller, *J. prakt. Chem.*, 1925, **109**, 142.

**1 : 2 : 3 : 4-Tetrahydronaphthalene-1 : 8-dicarboxylic Acid** (1 : 2 : 3 : 4-Tetrahydro-naphthalic acid).

Prisms. M.p. 196°. Sol. EtOH. Spar. sol. warm  $H_2O$ . Almost insol.  $Et_2O$ . Above m.p.  $\rightarrow$  anhydride.

*Di-Me ester*:  $C_{14}H_{16}O_4$ . MW, 248. Cryst. M.p. 74°.

*Di-Et ester*:  $C_{16}H_{20}O_4$ . MW, 276. M.p. 52°. B.p. 193°/17 mm.

*Anhydride*: cryst. from  $C_6H_6$ . M.p. 119°.

Cassares, Ranedo, *Chem. Abstracts*, 1923, **17**, 3030.

Willstätter, Jacquet, *Ber.*, 1918, **51**, 775.

**1 : 2 : 3 : 4-Tetrahydronaphthalene-2 : 3-dicarboxylic Acid.**

Plates. M.p. 199°. Very sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ . Sol. warm  $Et_2O$ . Spar. sol. cold  $H_2O$ . Above m.p.  $\rightarrow$  anhydride.

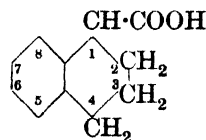
*Anhydride*: prisms from  $Et_2O$ . M.p. 184°. Mod. sol. EtOH,  $CHCl_3$ . Spar. sol.  $Et_2O$ . Insol. cold  $H_2O$ .

Baeyer, Perkin, *Ber.*, 1884, **17**, 450.

**Tetrahydronaphthalic Acid.**

See Tetrahydronaphthalene-1 : 8-dicarboxylic Acid.

**1 : 2 : 3 : 4-Tetrahydro-1-naphthoic Acid** (ac-Tetrahydro- $\alpha$ -naphthoic acid)



$C_{11}H_{12}O_2$

MW, 176

*d.*

Cryst. from pet. ether. M.p. 49–50°.  $[\alpha]_D + 14.01^\circ$  in  $CHCl_3$ .

*l.*

Plates from pet. ether. M.p. 52.5°.  $[\alpha]_D - 52.34^\circ$  in  $C_6H_6$ ,  $- 15.95^\circ$  in  $CHCl_3$ .

*dl.*

Prisms from AcOEt. M.p. 85°. Very sol. most org. solvents. Mod. sol. hot  $H_2O$ . Sol. 1052 parts cold  $H_2O$ .  $k = 4.45 \times 10^{-5}$  at 25°.

*Et ester*:  $C_{13}H_{16}O_2$ . MW, 204. B.p. 279°/749 mm.

*l-Menthyl ester*: b.p. 207°/10 mm.  $[\alpha]_D^{20} - 47.57^\circ$  in EtOH.

*Amide*:  $C_{11}H_{13}ON$ . MW, 175. Needles from EtOH. M.p. 116°.

Pickard, Yates, *J. Chem. Soc.*, 1906, **89**, 1102.

Kay, Morton, *J. Chem. Soc.*, 1914, **105**, 1571.

**5 : 6 : 7 : 8-Tetrahydro-1-naphthoic Acid** (ar-Tetrahydro- $\alpha$ -naphthoic acid).

Prisms from  $H_2O$ . M.p. 128°. Very sol. EtOH. Sol. hot  $H_2O$ . Spar. sol. cold  $H_2O$ .

*Et ester*: b.p. 156–9°/12 mm.

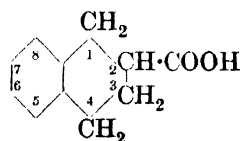
*Nitrile*:  $C_{11}H_{11}N$ . MW, 157. B.p. 277–9°/721 mm.

*Amide*: needles from  $H_2O$ . M.p. 182°. Sol. boiling  $H_2O$  and most org. solvents.

Bamberger, Bordt, *Ber.*, 1889, **22**, 628.

I.G., F.P., 649,626, (*Chem. Abstracts*, 1929, **23**, 2986).

**1 : 2 : 3 : 4-Tetrahydro-2-naphthoic Acid** (ac-Tetrahydro- $\beta$ -naphthoic acid)



$C_{11}H_{12}O_2$

MW, 176.

*d.*

Prisms from AcOH.Aq. M.p. 99°.  $[\alpha]_D + 40.35^\circ$  in  $CHCl_3$ .

*l.*

Prismatic needles from pet. ether. M.p. 99°.  $[\alpha]_D - 51.82^\circ$  in  $CHCl_3$ .

*dl.*

Needles from hot EtOH.Aq. M.p. 96°. Sol. 1661 parts  $H_2O$  at 14°.

*l-Menthyl ester*: b.p. 218°/11 mm.  $[\alpha]_D^{20} - 53.0^\circ$  in  $C_6H_6$ .

Pickard, Yates, *J. Chem. Soc.*, 1906, **89**, 1103.

Baeyer, Besemfelder, *Ann.*, 1891, **266**, 198.

**5 : 6 : 7 : 8-Tetrahydro-2-naphthoic Acid** (ar-Tetrahydro- $\beta$ -naphthoic acid).

Needles from EtOH. M.p. 154° (144°). B.p. 216°/14 mm. Insol. cold pet. ether.

*Chloride*:  $C_{11}H_{11}OCl$ . MW, 194.5. B.p. 162°/13 mm.

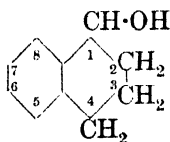
*Amide*:  $C_{11}H_{13}ON$ . MW, 175. Leaflets from EtOH-pet. ether. M.p. 137–8°.

*Anilide*: needles from EtOH. M.p. 147°.

v. Braun, Kirschbaum, Schumann, *Ber.*, 1920, **53**, 1161.

Coulson, *J. Chem. Soc.*, 1935, 80.

**1 : 2 : 3 : 4-Tetrahydro-1-naphthol** (ac-Tetrahydro- $\alpha$ -naphthol, 1-hydroxy-1 : 2 : 3 : 4-tetrahydronaphthalene, 1-hydroxytetralin)



$C_{10}H_{12}O$

MW, 148

*d.*

Pale yellow oil. B.p. 155–60°/25 mm.  $[\alpha]_D^{20} + 28.2^\circ$  in  $CHCl_3$ .

*dl.*

Thick oil. B.p. 264°/716 mm., 176.5–178°/53 mm., 132–4°/12–13 mm.  $D_4^{17}$  1.0896.  $n_D^{17}$  1.5671. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ .

*Acetyl*: b.p. 169°/34 mm., 105–10°/2 mm.

*Phenylurethane*: needles from ligroin. M.p. 121–2°.

Criegee, *Ann.*, 1930, **481**, 292.

Brochet, Cornubert, *Bull. soc. chim.*, 1922, **31**, 1280.

Strauss, Rohrbacher, *Ber.*, 1921, **54**, 57.

Bamberger, Lodter, *Ber.*, 1890, **23**, 197.

**5 : 6 : 7 : 8-Tetrahydro-1-naphthol** (ar-Tetrahydro- $\alpha$ -naphthol, 5-hydroxy-1 : 2 : 3 : 4-tetrahydronaphthalene, 5-hydroxytetralin, 1-tetralol).

Needles with strong phenolic odour. M.p. 68.5–69°. B.p. 264.5–265°. Very sol. most org. solvents. Less sol. hot  $H_2O$ .

*Et ether*:  $C_{12}H_{16}O$ . MW, 176. B.p. 259°/705 mm.

*Acetyl*: cryst. M.p. 73–5°.

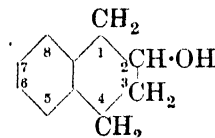
*Benzoyl*: m.p. about 46°.

Jacobsen, Turnbull, *Ber.*, 1898, **31**, 897.

Brochet, Cornubert, *Bull. soc. chim.*, 1922, **31**, 1280.

I.G., F.P., 644,408, (*Chem. Abstracts*, 1930, **24**, 862); U.S.P., 1,858,627, (*Chem. Abstracts*, 1932, **26**, 3808).

**1 : 2 : 3 : 4-Tetrahydro-2-naphthol** (ac-Tetrahydro- $\beta$ -naphthol, 2-hydroxy-1 : 2 : 3 : 4-tetrahydronaphthalene, 2-hydroxytetralin)



$C_{10}H_{12}O$

MW, 148

*d.*

Needles from pet. ether. M.p. 50°. B.p. 141°/17 mm.  $[\alpha]_D^{20} + 70.3^\circ$  in  $CHCl_3$ .

*Valeryl*: b.p. 169°/11 mm.  $D_4^{16}$  1.0317.

*Pelargonyl*: b.p. 195–7°/5 mm.  $D_4^{18}$  0.9821.

*Lauryl*: cryst. M.p. 35–6°. B.p. 218°/3 mm.  $D_4^{17}$  0.9677.

*Phenylurethane*: m.p. 119°.

*l.*

Needles from pet. ether. M.p. 50°. B.p. 141°/17 mm.  $D_4^{61}$  1.0589.  $[\alpha]_D^{20} - 67.1^\circ$  in  $CHCl_3$ .

*Acetyl*: b.p. 149°/18 mm.  $D_4^{14}$  1.0926.

*Propionyl*: b.p. 158–9°/18 mm.  $D_4^{16}$  1.0675.

*dl.*

Viscous oil. B.p. 264°/716 mm., 172.6–173.5°/53 mm., 144.5–146.5°/20 mm.  $D_4^{17}$  1.0715.  $n_D^{17}$  1.5523. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ . Turns brown in air.

*Acetyl*: b.p. 169°/34 mm.

*Benzoyl*: leaflets from EtOH. B.p. 254–5°/40 mm. Sol. hot EtOH, cold  $CHCl_3$ ,  $C_6H_6$ .

*Phenylurethane*: needles. M.p. 99°.

Bamberger, Lodter, *Ber.*, 1890, **23**, 197.

Pickard, Kenyon, *J. Chem. Soc.*, 1912, **101**, 1431.

Brochet, Cornubert, *Bull. soc. chim.*, 1922, **31**, 1280.

**5 : 6 : 7 : 8-Tetrahydro-2-naphthol** (ar-Tetrahydro- $\beta$ -naphthol, 6-hydroxy-1 : 2 : 3 : 4-tetrahydronaphthalene, 6-hydroxytetralin, 2-tetralol).

Needles from ligroin. M.p. 61.5–62.5° (58°). B.p. 275°/705 mm., 146°/13 mm. Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , hot ligroin. Spar. sol.  $H_2O$ . No col. with  $FeCl_3$ .

*Me ether*:  $C_{11}H_{14}O$ . MW, 162. B.p. 129–31°/11 mm.

*Et ether*:  $C_{12}H_{16}O$ . MW, 176. Oil with pleasant odour. B.p. 132–3°/15 mm., 129°/11 mm.  $D_4^{20}$  1.008.

*Acetyl*: viscous oil. B.p. 158°/14 mm.

*Benzoyl*: prisms. M.p. 96°. B.p. 220–2°/10 mm.

*p-Nitrobenzoyl*: m.p. 106.5°.

*Cinnamoyl*: m.p. 77.5°.

*Diphenylurethane*: m.p. 114°.

Schroeter, Schrauth, D.R.P., 299,603, (*Chem. Zentr.*, 1919, IV, 618).

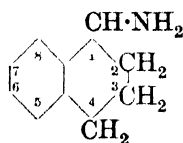
Schroeter *et al.*, *Ann.*, 1922, 426, 119.

Brochet, Cornubert, *Bull. soc. chim.*, 1922, 31, 1280.

Thoms, Kross, *Arch. Pharm.*, 1927, 265, 336.

I.G., F.P., 644,408, (*Chem. Abstracts*, 1930, 24, 862).

**1 : 2 : 3 : 4-Tetrahydro-1-naphthylamine**  
(*ac-Tetrahydro- $\alpha$ -naphthylamine*)



$C_{10}H_{13}N$

MW, 147

Oil with ammoniacal odour. B.p. 246.5°/714 mm. Sol. most org. solvents. Mod. sol. cold  $H_2O$ . Strong base.

*B, HNO<sub>2</sub>*: needles from  $H_2O$ . M.p. 138–9°.

*B<sub>2</sub>, H<sub>2</sub>PiCl<sub>6</sub>*: orange-yellow prisms + 2 $H_2O$ . M.p. 140°, anhyd. 190°.

*N-Acetyl*: needles from EtOH.Aq. M.p. 148–9°. Sol. EtOH, AcOH. Spar. sol. cold  $H_2O$ .

*N-2 : 4-Dinitrophenyl*: golden-yellow leaflets from EtOH. M.p. 121°.

Bamberger, Bamman, *Ber.*, 1889, 22, 964.

Green, Rowe, *J. Chem. Soc.*, 1918, 113, 957.

Komatsu, Amatatsu, *Chem. Abstracts*, 1931, 25, 500.

**5 : 6 : 7 : 8-Tetrahydro-1-naphthylamine**  
(*ar-Tetrahydro- $\alpha$ -naphthylamine*).

Oil. B.p. 275°, 233–5°/280 mm.  $D^{20}_D$  1.0625.  $n^{25}_D$  1.58964. Weak base. Reduces warm alc. Ag sols.

*N-Me*:  $C_{11}H_{15}N$ . MW, 161. Pale yellow liq. B.p. 150–2°/12 mm. *Picrate*: reddish-yellow cryst. powder. M.p. 174°.

*N-Di-Me*:  $C_{12}H_{17}N$ . MW, 175. B.p. 261–2°/721 mm., 131–131.5°/16 mm. Reduces Ag salts. *Methiodide*: prisms from  $H_2O$ . M.p. 164.5°. Sol. EtOH. Spar. sol.  $H_2O$ .

*N-Et*:  $C_{12}H_{17}N$ . MW, 175. B.p. 286–7°/717 mm. Sol. most org. solvents. Spar. sol.  $H_2O$ .

*N-2 : 4-Dinitrophenyl*: red leaflets from EtOH. M.p. 134°.

*N-Acetyl*: needles. M.p. 158°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

Bamberger, Althausse, *Ber.*, 1888, 21, 1789.

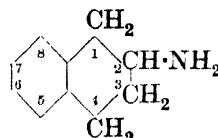
Bamberger, Helwig, *Ber.*, 1889, 22, 1315.

Bayer, D.R.P., 305,347, (*Chem. Zentr.*, 1918, I, 977).

Cassella, D.R.P., 479,401, (*Chem. Abstracts*, 1929, 23, 4710).

I.G., D.R.P., 581,831, (*Chem. Abstracts*, 1934, 28, 1059); Swiss P., 127,524. (*Chem. Abstracts*, 1929, 23, 1143).

**1 : 2 : 3 : 4 - Tetrahydro - 2 - naphthyl - amine**  
(*ac-Tetrahydro- $\beta$ -naphthylamine*)



$C_{10}H_{13}N$

MW, 147

*d.*

*B, HCl*: needles. M.p. 243–5°.  $[\alpha]^{12}_D + 71.9^\circ$  in  $H_2O$ .

*N-Acetyl*: needles from  $C_6H_6$ . M.p. 104–6°.  $[\alpha]^{16}_D + 36.9^\circ$  in  $C_6H_6$ .

*N-Benzoyl*: needles from  $Me_2CO$ . M.p. 155–7°.  $[\alpha]^{19}_D + 58^\circ$  in  $Me_2CO$ .

*l.*

*B, HCl*: cryst. from  $Me_2CO$ . M.p. 243–5°.  $[\alpha]^{19}_D - 69.7^\circ$  in  $H_2O$ .

*dl.*

B.p. 249°/710 mm. decomp., 140–140.5°/20 mm., 118.5°/8 mm.  $D^{22}_D$  1.0295.  $n^{22}_D$  1.56039. Sol. most org. solvents. Spar. sol. cold  $H_2O$ . Turns brown in air. Absorbs  $CO_2$ .

*B, HCl*: plates from  $H_2O$ . M.p. 237°.

*N-Me*:  $C_{11}H_{15}N$ . MW, 161. B.p. 118–119.8°/9 mm.  $D^{20}_D$  1.024. Sol. most org. solvents. Spar. sol.  $H_2O$ . *N-Acetyl*: yellowish syrup. B.p. 190–210°/17 mm. Sol. most org. solvents. Spar. sol.  $H_2O$ .

*N-Di-Me*:  $C_{12}H_{17}N$ . MW, 175. Oil with strong violet fluor. B.p. 132.3–133.3°/11 mm. Sol. most org. solvents. Spar. sol.  $H_2O$ . *B, HCl*: leaflets. M.p. 214–15° decomp. *Methiodide*: cryst. from EtOH. M.p. 228°.

*N-Et*:  $C_{12}H_{17}N$ . MW, 175. B.p. 267°/724 mm., 153°/23 mm. Volatile in steam. *B, HCl*: needles. M.p. 223.5°. *Picrate*: orange-red needles from  $H_2O$ . M.p. 183.5°. *N-Acetyl*: b.p. 328°/718 mm. Sol. most org. solvents. Insol.  $H_2O$ .

*N-Formyl*: needles from  $C_6H_6$ . M.p. 61°.

Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. cold H<sub>2</sub>O, ligroin.

N-Acetyl: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 107.5°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. hot H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O. Insol. ligroin.

N-Benzoyl: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 150-1°. Sol. hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. hot H<sub>2</sub>O.

Bamberger, Müller, *Ber.*, 1889, **22**, 1301.

Waser, *Ber.*, 1916, **49**, 1203.

Waser, Möllering, *Organic Syntheses*, Collective Vol., I, 486.

I.G., D.R.P., 581,831, (*Chem. Abstracts*, 1934, **28**, 1059).

### 5 : 6 : 7 : 8-Tetrahydro-2-naphthylamine (ar-Tetrahydro- $\beta$ -naphthylamine).

Needles from ligroin. M.p. 38°. B.p. 275-7°/713 mm. Sol. most org. solvents.

N-Me: b.p. 267.5°/210 mm. Sol. most org. solvents. Spar. sol. H<sub>2</sub>O.

N-Di-Me: b.p. 287°/718 mm., 168°/23 mm. Reduces Ag salts.

N-Et: b.p. 291-3°. Sol. most org. solvents. Spar. sol. H<sub>2</sub>O. Reduces hot NH<sub>3</sub>.AgNO<sub>3</sub>. B,HCl: needles. M.p. 173.5°.

N-Di-Et: b.p. 298°/709 mm., 167°/16 mm. Sol. most org. solvents. Spar. sol. H<sub>2</sub>O.

N-Acetyl: needles. M.p. 107°. Sol. EtOH.

N-Benzoyl: m.p. 167°.

Picrate: m.p. 204°.

Bamberger, Müller, *Ber.*, 1889, **22**, 1304.

Bamberger, Kitchelt, *Ber.*, 1890, **23**, 882.

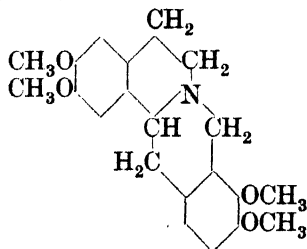
Cassella, D.R.P., 479,401, (*Chem. Abstracts*, 1929, **23**, 4710).

I.G., D.R.P., 581,831, (*Chem. Abstracts*, 1934, **28**, 1059).

### $\Delta^3$ -Tetrahydronicotinic Acid.

See Guvacine.

### Tetrahydropalmitine



C<sub>21</sub>H<sub>25</sub>O<sub>4</sub>N

d-.

MW, 355

Alkaloid from buds of *Corydalis cava*. M.p. 143°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 291° in EtOH. Turns yellow in air.

B,HCl: m.p. 266°.

l-.

M.p. 141-2°. [ $\alpha$ ]<sub>D</sub><sup>14</sup> - 290.8° in EtOH.

dl-.

B,HCl: needles from H<sub>2</sub>O. M.p. 215°.

B,HI: orange-yellow needles. M.p. 241° decomp.

B,HAuCl<sub>4</sub>: plates from EtOH.Aq. M.p. 201°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: orange cryst. powder. M.p. 228° decomp.

• Methiodide: exists in two forms. (i) Cryst. from MeOH. M.p. 230°. (ii) M.p. 266° decomp.

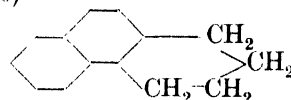
Späth, Mosettig, Tröthandl, *Ber.*, 1923, **56**, 875.

Späth, Mosettig, *Ber.*, 1926, **59**, 1496.

Späth, Leithe, *Ber.*, 1930, **63**, 3007.

Haworth, Koepfli, Perkin, *J. Chem. Soc.*, 1927, 553.

### 1 : 2 : 3 : 4 - Tetrahydrophenanthrene (Tetanthere)



C<sub>14</sub>H<sub>14</sub>

MW, 182

Cryst. from MeOH. M.p. 33-4°. B.p. 173°/11 mm. D<sub>4</sub><sup>20</sup> 1.0601. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>, ligroin. Sol. 15 parts MeOH.

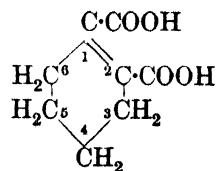
Picrate: reddish-yellow needles from EtOH. M.p. 111°.

Schroeter, Müller, Huang, *Ber.*, 1929, **62**, 652.

### Tetrahydrophenol.

See Cyclohexenol.

### $\Delta^1$ -Tetrahydrophthalic Acid (Cyclohexene-1 : 2-dicarboxylic acid)



C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>

MW, 170

Plates from H<sub>2</sub>O. Heat  $\rightarrow$  anhydride before melting. Boil with alkalis  $\rightarrow$   $\Delta^2$ -tetrahydrophthalic acid. Alk. KMnO<sub>4</sub>  $\rightarrow$  adipic acid.

Di-Et ester: C<sub>12</sub>H<sub>18</sub>O<sub>4</sub>. MW, 226. B.p. 160°/14 mm., 147°/10 mm. D<sub>4</sub><sup>20</sup> 1.0803. n<sub>D</sub><sup>20</sup> 1.47466.

Monoamide: C<sub>8</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 169. Needles from EtOH. Decomp. at 170°  $\rightarrow$   $\Delta^1$ -tetrahydrophthalimide.

Anhydride: C<sub>8</sub>H<sub>6</sub>O<sub>3</sub>. MW, 152. Plates from Et<sub>2</sub>O. M.p. 74°. Sol. Et<sub>2</sub>O.

Imide: C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 151. Cryst. from

EtOH, Et<sub>2</sub>O, or ligroin. M.p. 169–70°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>.

*Anil*: leaflets or needles from AcOH. M.p. 137°. Sol. EtOH, AcOH, Me<sub>2</sub>CO.

*Monoanilide*: cryst. M.p. 155° (rapid heat.). Sol. EtOH, Me<sub>2</sub>CO. Spar. sol. C<sub>6</sub>H<sub>6</sub>. FeCl<sub>3</sub>  $\longrightarrow$  pale green col.

Kon, Nandi, *J. Chem. Soc.*, 1933, 1633.

Hückel, Lampert, *Ber.*, 1934, 67, 1812.

Baeyer, *Ann.*, 1890, 258, 203.

Küster, *Z. physiol. Chem.*, 1908, 55, 520.

**$\Delta^2$ -Tetrahydrophthalic Acid** (*Cyclohexene-1:6-dicarboxylic acid*).

Prisms. M.p. 215° (rapid heat.). Sol. 114 parts H<sub>2</sub>O at 10°.  $k$  (first) =  $7.4 \times 10^{-5}$  at 25°; (second) =  $3.2 \times 10^{-7}$  at 100°. Above 220°  $\longrightarrow$  anhydride.

*Di-Et ester*: b.p. 155°/12 mm.  $D_4^{20}$  1.0760.  $n_D$  1.4700.

*Dichloride*: C<sub>8</sub>H<sub>8</sub>O<sub>2</sub>Cl<sub>2</sub>. MW, 207. Yellow oil. B.p. 129°/14 mm.

*Anhydride*: prisms from Et<sub>2</sub>O. M.p. 78–9°.

*Imide*: m.p. 172–3°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, Me<sub>2</sub>CO. Insol. cold H<sub>2</sub>O.

See first reference above and also

Küster, *Z. physiol. Chem.*, 1908, 55, 520.

Baeyer, *Ann.*, 1890, 258, 199.

Kaufmann, Voss, *Ber.*, 1923, 56, 2513.

**$\Delta^3$ -Tetrahydrophthalic Acid** (*Cyclohexene-3:4-dicarboxylic acid*).

Free acid not isolated.  $k = 5.81 \times 10^{-4}$  at 25°.

*Anhydride*: leaflets. M.p. 70°. Sol. EtOH. Spar. sol. H<sub>2</sub>O.

*p-Methoxyanil*: leaflets. M.p. 88°.

Abati, de Bernardinis, *Gazz. chim. ital.*, 1906, 36, ii, 824.

**$\Delta^4$ -Tetrahydrophthalic Acid** (*Cyclohexene-4:5-dicarboxylic acid*).

*Cis*:

Prisms from H<sub>2</sub>O. M.p. 174° (166°). Sol. 108 parts H<sub>2</sub>O at 6°.

*Anhydride*: cryst. from ligroin. M.p. 103–4° (58–9°). Sol. usual org. solvents. Spar. sol. ligroin, pet. ether.

*Trans*:

*d.*

Powder. M.p. 165°.  $[\alpha]_D^{25} + 115.2^\circ$ .

*Anhydride*: leaflets. M.p. 128°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>.  $[\alpha]_D^{25} + 6.6^\circ$  in EtOH.

*l.*

Powder. M.p. 167°. Mod. sol. EtOH.  $[\alpha]_D^{25} - 97.4^\circ$ .

*dl.*

Leaflets from H<sub>2</sub>O. M.p. 215–18°. Sol. 690 parts H<sub>2</sub>O at 6°.

*Di-Me ester*: C<sub>10</sub>H<sub>14</sub>O<sub>4</sub>. MW, 198. M.p. 39–40°.

*Anhydride*: cryst. from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 130° (141°).

Diels, Alder, *Ber.*, 1929, 62, 2087; *Ann.*, 1928, 460, 113.

Abati, de Horatio, *Gazz. chim. ital.*, 1909, 39, i, 558.

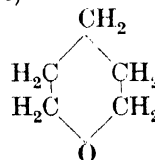
Baeyer, *Ann.*, 1892, 269, 203.

I.G., U.S.P., 1,944,731, (*Chem. Abstracts*, 1934, 28, 2016).

**1:4:5:6-Tetrahydro- $\alpha$ -picoline.**

6-Methyl-1:2:3:4-tetrahydropyridine, *q.v.*

**Tetrahydropyran** (*Pentamethylene oxide, pyran tetrahydride*)



C<sub>5</sub>H<sub>10</sub>O

MW, 86

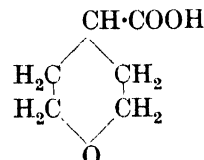
Liq. with characteristic odour. B.p. 88°/760 mm.  $D_0^{15.5}$  0.883,  $D_4^{15}$  0.8855.  $n_D^{15.5}$  1.4195. Volatile in steam.

Clarke, *J. Chem. Soc.*, 1912, 101, 1802.

Paul, *Bull. soc. chim.*, 1933, 53, 1493.

Allen, Hibbert, *J. Am. Chem. Soc.*, 1934, 56, 1398.

**Tetrahydropyran-4-carboxylic Acid**



C<sub>6</sub>H<sub>10</sub>O<sub>3</sub>

MW, 130

Cryst. from Et<sub>2</sub>O. M.p. 87°. Sol. H<sub>2</sub>O. Spar. sol. pet. ether.

*Me ester*: C<sub>7</sub>H<sub>12</sub>O<sub>3</sub>. MW, 144. Liq. with pleasant odour. B.p. 80.5–81°/16 mm.

*Et ester*: C<sub>8</sub>H<sub>14</sub>O<sub>3</sub>. MW, 158. B.p. 82.5°/12 mm.

*Chloride*: C<sub>6</sub>H<sub>9</sub>O<sub>2</sub>Cl. MW, 148.5. B.p. 85–6°/16 mm.

*Amide*: C<sub>6</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 129. Plates from EtOH. M.p. 179°.

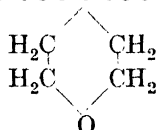
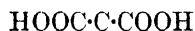
*Nitrile*: C<sub>6</sub>H<sub>9</sub>ON. MW, 111. B.p. 82–3°/10 mm. Very sol. H<sub>2</sub>O.

*Anilide* : plates from EtOH. M.p. 163°. Almost insol. Et<sub>2</sub>O.

Gibson, Johnson, *J. Chem. Soc.*, 1930, 2527.

v. Braun, Kohler, *Ber.*, 1917, 50, 1658.

### Tetrahydropyran-4 : 4-dicarboxylic Acid



C<sub>7</sub>H<sub>10</sub>O<sub>5</sub> MW, 174

Leaflets from Et<sub>2</sub>O-pet. ether. M.p. 172-3°. Sol. Et<sub>2</sub>O. Spar. sol. pet. ether. Above m.p. → tetrahydropyran-4-carboxylic acid.

*Di-Et ester* : C<sub>11</sub>H<sub>18</sub>O<sub>5</sub>. MW, 230. B.p. 152-5°/21 mm., 134-5°/12 mm.

*Mononitrile* : C<sub>7</sub>H<sub>9</sub>O<sub>3</sub>N. MW, 155. Prisms from H<sub>2</sub>O. M.p. 160-2°. Very sol. hot H<sub>2</sub>O. *Et ester* : C<sub>9</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 183. B.p. 125°/16 mm. *Amide* : C<sub>7</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>. MW, 154. Plates from EtOH. M.p. 158°.

See previous references.

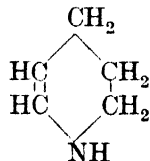
### Tetrahydropyreneolone.

See 4-Methyl-3-*n*-amylcyclopentanolone-2.

### Tetrahydropyreneone.

See 3-Methyl-2-*n*-amylcyclopentanone.

### 1 : 2 : 3 : 4-Tetrahydropyridine (Δ<sup>2</sup>-Piperidine)



C<sub>5</sub>H<sub>9</sub>N MW, 83

Decomp. on boiling with formation of a dimer. *B, HCl* : m.p. 230°.

*B, HBr* : m.p. 178°.

*Acetyl* : b.p. 219.5-220.5°. D<sub>4</sub><sup>16.5</sup> 1.0531. Sol. usual org. solvents.

*Dimer* : cryst. M.p. 61°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>. Heat of comb. C<sub>v</sub> 1532.7 Cal. Reduces Ag salts.

Lellmann, Schwaderer, *Ber.*, 1889, 22, 1320.

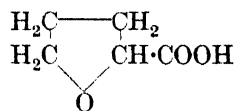
Wolffenstein, *Ber.*, 1892, 25, 2782.

Paal, Hubaleck, *Ber.*, 1901, 34, 2761.

### Δ<sup>3</sup> - Tetrahydropyridine - 3 - carboxylic Acid.

See Guvacine.

### Tetrahydropyromucic Acid (*Tetrahydrofuran-2-carboxylic acid, tetrahydro-β-furoic acid*)



C<sub>5</sub>H<sub>8</sub>O<sub>3</sub>

MW, 116

Cryst. M.p. 21°. B.p. 145°/25 mm., 131°/14 mm. D<sub>20</sub><sup>20</sup> 1.1933. n<sub>D</sub><sup>19</sup> 1.4585. k = 1.4 × 10<sup>-4</sup> at 25°.

*Et ester* : C<sub>7</sub>H<sub>12</sub>O<sub>3</sub>. MW, 144. Liq. with pleasant fruity odour. B.p. 82°/11 mm. D<sub>20</sub><sup>20</sup> 1.0792. n<sub>D</sub><sup>18</sup> 1.4445. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Amide* : C<sub>5</sub>H<sub>9</sub>O<sub>2</sub>N. MW, 115. Leaflets from Et<sub>2</sub>O. M.p. 80°. B.p. 135-40°/20 mm. Sol. H<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O.

*Nitrile* : C<sub>5</sub>H<sub>7</sub>ON. MW, 97. B.p. 80-2°/23 mm. n<sub>D</sub><sup>25</sup> 1.4351.

Wienhaus, Sorge, *Ber.*, 1913, 46, 1929.

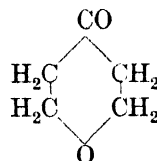
Kaufmann, Adams, *J. Am. Chem. Soc.*, 1923, 45, 3041.

Williams, *Ber.*, 1927, 60, 2512.

### Tetrahydro-α-pyrone.

See δ-Valerolactone.

### Tetrahydro-γ-pyrone



C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>

MW, 100

B.p. 163-6°/742 mm., 67-9°/18 mm. Misc. with H<sub>2</sub>O. D<sub>24.5</sub><sup>24.5</sup> 1.0795. n<sub>D</sub><sup>24.5</sup> 1.4529.

*Oxime* : cryst. from EtOH. M.p. 87-8°.

*Phenylsemicarbazone* : needles from EtOH.Aq. M.p. 169°.

Borsche, *Ber.*, 1915, 48, 683.

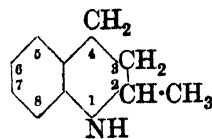
Borsche, Thiele, *Ber.*, 1923, 56, 2012.

Cornubert, Robinet, *Bull. soc. chim.*, 1933, 53, 565.

### Tetrahydropyrrole.

See Pyrrolidine.

### 1 : 2 : 3 : 4-Tetrahydroquinaldine (2-Methyl-1 : 2 : 3 : 4-tetrahydroquinoline)



C<sub>10</sub>H<sub>13</sub>N

MW, 147

d-.

Oil.  $[\alpha]_D^{20} + 58.1^\circ$ .*B.HCl*: plates +  $H_2O$  from EtOH. M.p. 196.5–197.5°.  $[\alpha]_D^{21.4} + 66.1^\circ$  in  $H_2O$ .*N-Et*:  $C_{12}H_{17}N$ . MW, 175. B.p. 256°.  $D_4^{20}$  0.9942.  $[\alpha]_D^{20} + 12.1^\circ$ .*N-Benzoyl*: cryst.  $[\alpha]_D^{18} - 247^\circ$  in  $C_6H_6$ .

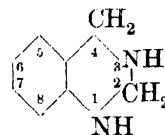
l-.

B.p. 158–9°/59 mm.  $D_4^{18}$  1.0207.  $n_D^{24}$  1.5705. Misc. with usual org. solvents.*B.HCl*: plates +  $H_2O$  from EtOH. M.p. 196.5–197.5°.  $[\alpha]_D^{19} - 60.4^\circ$  in  $H_2O$ .*N-Benzoyl*: cryst. from EtOH. M.p. 117.5–118°. Sol.  $C_6H_6$ . Mod. sol. EtOH.  $[\alpha]_D^{25} + 324^\circ$  in  $C_6H_6$ .*N-o-Nitrobenzoyl*: yellow needles from EtOH. M.p. 110°. Sol. EtOH,  $Me_2CO$ , AcOH,  $C_6H_6$ .  $[\alpha]_D^{20} + 17.1^\circ$  in EtOH.*N-m-Nitrobenzoyl*: needles from EtOH. M.p. 97°.  $[\alpha]_D^{20} + 241.6^\circ$  in EtOH.*N-p-Nitrobenzoyl*: yellowish cryst. from  $C_6H_6$ -pet. ether. M.p. 110°.  $[\alpha]_D^{20} + 369.6^\circ$  in EtOH.*N-p-Toluenesulphonyl*: cryst. from EtOH. M.p. 109°.  $[\alpha]_D^{20} - 137^\circ$  in EtOH.*Picrate*: yellow plates of needles from EtOH. M.p. 148–50°.  $[\alpha]_D^{20} - 33.0^\circ$  in EtOH.

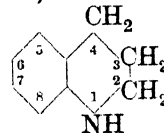
dl-.

B.p. 246–8°/709 mm., 115–16°/12 mm.  $D_4^{18}$  1.0208.  $n_D^{22}$  1.5727. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ . Heat of comb.  $C_v$  1380.6 Cal.*B.HCl*: m.p. 190° (128–30°).*N-Me*:  $C_{11}H_{15}N$ . MW, 161. B.p. 247–8°, 144°/28 mm.  $n_D^{19}$  1.5678. Turns brown in air.*Methiodide*: needles from EtOH. M.p. 205°.*N-Et*: b.p. 256°.*N-Benzoyl*: cryst. from AcOEt. M.p. 119.2° (116°). Sol. most org. solvents.*N-m-Nitrobenzoyl*: pale yellow leaflets. M.p. 114°.*Picrate*: prisms from EtOH. M.p. 153–4°.Pope, Peachey, *J. Chem. Soc.*, 1899, 75, 1082.Pope, Winmill, *J. Chem. Soc.*, 1912, 101, 2311.v. Braun, Gmelin, Schultheiss, *Ber.*, 1923, 56, 1344.Tröger, Ungar, *J. prakt. Chem.*, 1926, 112, 251.**5 : 6 : 7 : 8-Tetrahydroquinaldine** (2-Methyl-5 : 6 : 7 : 8-tetrahydroquinoline).B.p. 225°/762 mm., 101–4°/12 mm.  $D^{18}$  1.0000.  $n_D^{23}$  1.5310.*B.HCl*: m.p. 164°.*B\_2H\_2PtCl\_6*: m.p. 198°.*Methiodide*: m.p. 118°. Hygroscopic.*Picrate*: lemon-yellow cryst. M.p. 157° (154°).See last two references above and also Basu, *Ann.*, 1934, 512, 131.**Tetrahydroquinaldinic Acid.**

See Tetrahydroquinoline-2-carboxylic Acid.

**1 : 2 : 3 : 4-Tetrahydroquinazoline** $C_8H_{10}N_2$ 

MW, 134

Leaflets from  $C_6H_6$ -ligroin, m.p. 78–9°: cryst. +  $H_2O$  from  $H_2O$ , m.p. 49–51°.*B.HCl*: needles from EtOH. M.p. 193–5°.*B\_2H\_2PtCl\_6*: orange-yellow needles. Does not melt below 270°.*3-N-Allyl*: oil. B.p. 270–2°.*3-N-Phenyl*: needles from EtOH. M.p. 119°.*3-N-o-Tolyl*: cryst. from  $Et_2O$ . M.p. 140°.*3-N-p-Tolyl*: needles from EtOH. M.p. 127°.Busch, Dietz, *J. prakt. Chem.*, 1896, 53, 418.Busch, *J. prakt. Chem.*, 1895, 51, 129.Gabriel, *Ber.*, 1903, 36, 811.**1 : 2 : 3 : 4-Tetrahydroquinoline** (Py-Tetrahydroquinoline) $C_9H_{11}N$ 

MW, 133

B.p. 249–50°/755 mm.  $D_4^{23.9}$  1.0546. Heat of comb.  $C_v$  1226.6 Cal.  $n_D^{23.9}$  1.5933.*B.HCl*: prisms from EtOH. M.p. 180–1°.*B.HBr*: m.p. 167°.*B.HI*: needles from MeOH. M.p. 170°.*N-Allyl*: b.p. 264–6°/755 mm. *B.HI*: m.p. 141°.*N-Benzyl*: needles from EtOH. M.p. 36–7°. B.p. 218–22°/38 mm.*N-o-Nitrobenzyl*: m.p. 111°.*N-m-Nitrobenzyl*: red prisms from EtOH. M.p. 99°.*N-p-Nitrobenzyl*: light red cryst. from EtOH. M.p. 102°.*N-Acetyl*: b.p. 295°.*N-Benzoyl*: plates from EtOH. M.p. 76°.*N-Me*: see Kairolin.



N-*Et*: see Kairolin A.

Hoffmann, Koenigs, *Ber.*, 1883, 16, 728.

Wedekind, *Ber.*, 1902, 35, 185.

Lellmann, Pekrun, *Ann.*, 1890, 259, 50.

Skita, Meyer, *Ber.*, 1912, 45, 3594.

**5 : 6 : 7 : 8-Tetrahydroquinoline** (Bz-Tetrahydroquinoline).

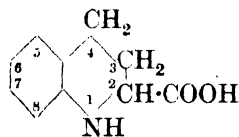
B.p. 222°.  $D_4^{25}$  1.025. No col. with  $\text{FeCl}_3$ .

$\text{B}_2\text{H}_2\text{PtCl}_6$ : yellow cryst. Decomp. at 210°.

Picrate: m.p. 157°.

v. Braun, Lemke, *Ann.*, 1930, 478, 190.

**1 : 2 : 3 : 4-Tetrahydroquinoline-2-carboxylic Acid** (1 : 2 : 3 : 4-Tetrahydroquinaldinic acid)



$\text{C}_{10}\text{H}_{11}\text{O}_2\text{N}$

MW, 177

Brownish-yellow cryst. from  $\text{C}_6\text{H}_6$ . M.p. 112–13°.

*B, HCl*: cryst. +  $2\text{H}_2\text{O}$  from  $\text{HCl.Aq.}$  M.p. 115–20°, anhyd. 200° decomp.

*Me ester*:  $\text{C}_{11}\text{H}_{13}\text{O}_2\text{N}$ . MW, 191. B.p. 180°/15 mm. *B, HCl*: cryst. from  $\text{MeOH-Et}_2\text{O}$ . M.p. 191°.

N-*Nitroso*: needles from  $\text{EtOH}$ ,  $\text{AcOEt}$  or  $\text{C}_6\text{H}_6$ . Decomp. at 132°.

N-*Acetyl*: prisms. M.p. 175–6°.

N-*Benzoyl*: m.p. 187–8°.

Wieland, Hetteche, Hoshino, *Ber.*, 1928, 61, 2377.

**1 : 2 : 3 : 4-Tetrahydroquinoline-4-carboxylic Acid** (1 : 2 : 3 : 4-Tetrahydrocinchoninic acid).

Free acid not isolated.

*B, HCl*: prisms +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ .  $\text{FeCl}_3 \rightarrow$  brownish-green col.  $\rightarrow$  green on standing.

*Me ester*: cryst. from pet. ether. M.p. 88°. N-*Nitroso*: reddish-yellow cryst. powder. M.p. 69–70°.

N-*Me*: kairolin-4-carboxylic acid.  $\text{C}_{11}\text{H}_{13}\text{O}_2\text{N}$ . MW, 191. Prisms +  $2\text{H}_2\text{O}$  from  $\text{EtOH}$ . M.p. 169–70° decomp. Very sol.  $\text{EtOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Bitter taste. *Anhydride*: oil. B.p. 279°/744 mm. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

N-*Acetyl*: cryst. from  $\text{H}_2\text{O}$ . M.p. 164.5°. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ .

N-*Nitroso*: yellowish needles from  $\text{H}_2\text{O}$ . M.p. 137°. Sol.  $\text{EtOH}$ , hot  $\text{H}_2\text{O}$ . Less sol.  $\text{Et}_2\text{O}$ .

Weidel, *Monatsh.*, 1882, 3, 61.

Weidel, Hazura, *Monatsh.*, 1884, 5, 643.

v. Braun, Lemke, *Ann.*, 1930, 478, 194.

**5 : 6 : 7 : 8-Tetrahydroquinoline-4-carboxylic Acid** (5 : 6 : 7 : 8-Tetrahydrocinchoninic acid).

Cryst. from  $\text{H}_2\text{O}$ . M.p. 242° decomp. Sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ . Long heating  $\rightarrow$  5 : 6 : 7 : 8-tetrahydroquinoline.

v. Braun, Lemke, *Ann.*, 1930, 478, 190.

**1 : 2 : 3 : 4-Tetrahydroquinoline-5-carboxylic Acid.**

Needles or leaflets from  $\text{EtOH.Aq.}$  M.p. 146–7°.

N-*Me*: kairolin-5-carboxylic acid. Needles from  $\text{EtOH.Aq.}$  M.p. 164°. Sol.  $\text{EtOH}$ . Less sol.  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

N-*Nitroso*: yellow prisms from  $\text{EtOH.Aq.}$  M.p. 186° decomp.

Fischer, Körner, *Ber.*, 1884, 17, 765.

Lellmann, Alt, *Ann.*, 1887, 237, 315.

**1 : 2 : 3 : 4-Tetrahydroquinoline-6-carboxylic Acid.**

Needles. Decomp. about 170°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

N-*Me*: kairolin-6-carboxylic acid. Cryst. from  $\text{C}_6\text{H}_6$ . M.p. about 224° decomp. Sol.  $\text{EtOH}$ . Less sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ .

N-*Et*: leaflets from  $\text{C}_6\text{H}_6$ . M.p. about 200° decomp.

N-*Acetyl*: needles from  $\text{H}_2\text{O}$ . M.p. 187°. Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Less sol. hot  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ , ligroin.

N-*Nitroso*: yellowish prisms. from  $\text{C}_6\text{H}_6$ . Decomp. about 181°.

Fischer, Endres, *Ber.*, 1902, 35, 2613.

Kunckell, Vollhase, *Ber.*, 1909, 42, 3198.

**1 : 2 : 3 : 4-Tetrahydroquinoline-7-carboxylic Acid.**

Leaflets from  $\text{EtOH.Aq.}$  M.p. 189°. Sol.  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Less sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ .

N-*Me*: kairolin-7-carboxylic acid. Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 185°.

N-*Et*: prisms from  $\text{C}_6\text{H}_6$ . M.p. 163–4°.

N-*Nitroso*: yellowish prisms from  $\text{C}_6\text{H}_6$ . Decomp. at 191°.

Fischer, Endres, *Ber.*, 1902, 35, 2612.

**1 : 2 : 3 : 4-Tetrahydroquinoline-8-carboxylic Acid.**

Needles from ligroin or  $\text{EtOH.Aq.}$  M.p. 163°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ . Very spar. sol. hot  $\text{H}_2\text{O}$ . Neutral or alkaline sols. show blue fluor. Sublimes.

N-Me: kairoline-8-carboxylic acid. Needles from  $C_6H_6$ . M.p. 218–19°.

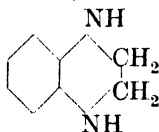
N-Et: leaflets from EtOH.Aq. M.p. 196–7°.

N-Nitroso: plates from  $C_6H_6$ . Decomp. about 124°.

Fischer, Endres, *Ber.*, 1902, **35**, 2611.

Tafel, *Ber.*, 1894, **27**, 825.

1 : 2 : 3 : 4-Tetrahydroquinoxaline (Ethylene-o-phenylenediamine)



$C_8H_{10}N_2$

MW, 134

Leaflets from  $H_2O$ ,  $Et_2O$  or pet. ether. M.p. 96.5–97°. B.p. 288.5–289.5°. Very sol.  $Et_2O$ . Sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ , hot  $H_2O$ . Spar. sol. boiling pet. ether. Alk.  $K_3Fe(CN)_6 \rightarrow$  quinoxaline.  $FeCl_3 \rightarrow$  violet col. with conc. sols., blue with dilute.

N-Me:  $C_9H_{12}N_2$ . MW, 148. Pale yellow oil. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ , pet. ether. Spar. sol. boiling  $H_2O$ .  $FeCl_3 \rightarrow$  blue col.

N : N'-Diacyl: cryst. from  $Et_2O$ . M.p. 144°. B.p. 350° decomp. Sol.  $H_2O$ . Mod. sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ .

N : N'-Dibenzenesulphonyl: cryst. from AcOH. M.p. 180°.

N : N'-Dinitroso: pale yellow microneedles from  $CHCl_3$ -ligroin. M.p. 168° decomp.

I.G., D.R.P., 495,101, (*Chem. Abstracts*, 1930, **24**, 3251).

Hinsberg, Strupler, *Ann.*, 1895, **287**, 225.

Ris, *Ber.*, 1888, **21**, 378.

Merz, Ris, *Ber.*, 1887, **20**, 1191, 1196.

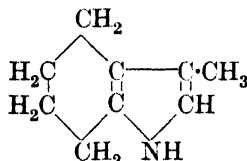
#### Tetrahydrosantonin.

See Santonan.

#### Tetrahydrosantoninic Acid.

See Santonanic Acid.

4 : 5 : 6 : 7-Tetrahydroskatole (3-Methyl-4 : 5 : 6 : 7-tetrahydroindole)



$C_9H_{13}N$

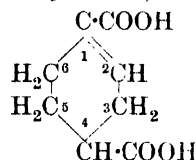
MW, 135

B.p. 105°/12 mm.  $D_4^{14}$  0.9698.

v. Braun, Bayer, Blessing, *Ber.*, 1924, **57**, 400.

Dict. of Org. Comp.—III.

$\Delta^1$ -Tetrahydroterephthalic Acid (Cyclohexene-1 : 4-dicarboxylic acid)



$C_8H_{10}O_4$

MW, 170

Prisms from  $H_2O$ . Does not melt below 300°. Sol. 4066 parts cold  $H_2O$ . Sublimes.  $k$  (first) =  $5.0 \times 10^{-5}$  at 25°; (second) =  $2.8 \times 10^{-6}$  at 100°. Heat of comb.  $C_p$  882.8 Cal. Alk.  $KMnO_4 \rightarrow$  oxalic acid.

Di-Me ester:  $C_{10}H_{14}O_4$ . MW, 198. Needles from  $H_2O$ . M.p. 39° (37°). B.p. 153.3–154.5°/20 mm., 147°/9 mm. Very sol. most solvents. Heat of comb.  $C_p$  1226 Cal.

Di-l-menthyl ester: needles from EtOH. M.p. 125°.  $[\alpha]_D^{20} - 69.4^\circ$  in  $CHCl_3$ .

Diphenyl ester: plates from AcOEt or  $Me_2CO$ . M.p. 145°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , ligroin.

Rupe, *Ann.*, 1910, **373**, 123.

Baeyer, *Ann.*, 1888, **245**, 159.

$\Delta^2$ -Tetrahydroterephthalic Acid (Cyclohexene-3 : 6-dicarboxylic acid).

Cis:

Plates from  $H_2O$ . M.p. 150.5°. Sol. 37 parts cold  $H_2O$ .

Trans:

Rhomboheda from  $H_2O$ . M.p. 220°. Sol. 588 parts cold  $H_2O$ . Very sol. hot  $H_2O$ .

Di-Me ester: plates or prisms from ligroin. M.p. 3°.

Diphenyl ester: plates from  $C_6H_6$ -ligroin. M.p. 107°.

Dibenzyl ester: cryst. from EtOH. M.p. 48°.

See first reference above and also

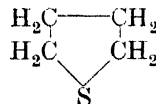
Baeyer, *Ann.*, 1889, **251**, 279, 306.

Baeyer, Herb, *Ann.*, 1890, **258**, 39.

#### Tetrahydrothiazine.

See 1 : 4-Thiazan.

Tetrahydrothiophene (Tetramethylene sulphide)



$C_4H_8S$

MW, 88

Mobile liq. with penetrating odour. B.p. 118–19°.  $D_4^{18}$  0.9607.  $n_D^{18}$  1.4871. Volatile in steam. Misc. with most solvents except  $H_2O$ .  $KMnO_4 \rightarrow$  sulphone, m.p. 20–1° (8–10°), b.p.

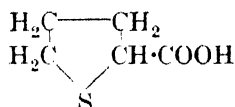
285–8°/743 mm., 153–4°/18 mm., 149.5–150°/15 mm. Forms add. comp. with one mol.  $\text{HgCl}_2$ , needles, m.p. 124.5–125.5°.

Grischkewitsch-Trochimowski, *J. Russ. Phys.-Chem. Soc.*, 1916, **48**, 901.

Bost, Conn, *Chem. Abstracts*, 1933, **27**, 5323.

Backer, Bolt, *Rec. trav. chim.*, 1935, **54**, 539.

### Tetrahydrothiophene-2-carboxylic Acid



$\text{C}_5\text{H}_8\text{O}_2\text{S}$

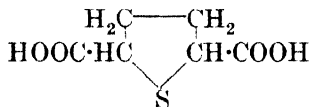
MW, 132

Plates from  $\text{H}_2\text{O}$ . M.p. 51°. Very sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Heat of comb.  $\text{C}_v$  753.3 Cal.  $k = 1.15 \times 10^{-4}$  at 25°. Volatile in steam. Reduces boiling  $\text{NH}_3 \cdot \text{AgNO}_3$ .

*Me ester*:  $\text{C}_6\text{H}_{10}\text{O}_2\text{S}$ . MW, 146. B.p. 206°.

Ernst, *Ber.*, 1887, **20**, 518.

### Tetrahydrothiophene-2 : 5-dicarboxylic Acid



$\text{C}_6\text{H}_8\text{O}_4\text{S}$

MW, 176

Plates. M.p. 162°. Sol.  $\text{H}_2\text{O}$ . Less sol.  $\text{Et}_2\text{O}$ . Reduces warm  $\text{NH}_3 \cdot \text{AgNO}_3$ .

Ernst, *Ber.*, 1886, **19**, 3275.

### Tetrahydrothiopyran.

See Pentamethylene sulphide.

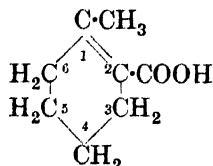
### Tetrahydrothiotolene.

See Methyltetrahydrothiophene.

### Tetrahydrotoluene.

See Methylcyclohexene.

### $\Delta^1$ -Tetrahydro-*o*-toluic Acid (2-Methylcyclohexene-1-carboxylic acid)



$\text{C}_8\text{H}_{12}\text{O}_2$

MW, 140

Needles from  $\text{H}_2\text{O}$ . M.p. 87° (79–80°). Sol. EtOH,  $\text{Me}_2\text{CO}$ , pet. ether. Spar. sol.  $\text{H}_2\text{O}$ . Ox.  $\rightarrow$  3-acetobutyric acid.

*Et ester*:  $\text{C}_{10}\text{H}_{16}\text{O}_2$ . MW, 168. B.p. 148°/100 mm.

Kay, Perkin, *J. Chem. Soc.*, 1905, **87**, 1068.

Mazza, di Mase, *Gazz. chim. ital.*, 1927, **57**, 300.

### $\Delta^2$ -Tetrahydro-*o*-toluic Acid (6-Methylcyclohexene-1-carboxylic acid).

Cryst. from pet. ether. M.p. 80°.

Mazza, Cremona, *Gazz. chim. ital.*, 1927, **57**, 318.

### $\Delta^3$ -Tetrahydro-*o*-toluic Acid (4-Methylcyclohexene-3-carboxylic acid).

*Cis*:

B.p. 145–6°/20 mm. Ox.  $\rightarrow$  *trans*-form.

*Et ester*: b.p. 143–4°/100 mm.

*Trans*:

M.p. 60–2°. B.p. 162–3°/50 mm.

*Et ester*: b.p. 144–6°/100 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 744.

### $\Delta^4$ -Tetrahydro-*o*-toluic Acid (5-Methylcyclohexene-4-carboxylic acid).

Thick syrup. B.p. 180–1°/100 mm., 143°/20 mm., 135°/12 mm. Ox.  $\rightarrow$  methylbutane-1 : 2 : 4-tricarboxylic acid.

*Et ester*: b.p. 140°/100 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 754.

### $\Delta^5$ -Tetrahydro-*o*-toluic Acid (3-Methylcyclohexene-4-carboxylic acid).

B.p. 139°/20 mm., 114–30°/9 mm.

*Et ester*: b.p. 204–8°, 138–9°/100 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 735.

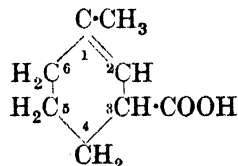
Skita, Ardan, Krauss, *Ber.*, 1908, **41**, 2944.

### $\Delta^6$ -Tetrahydro-*o*-toluic Acid (2-Methylcyclohexene-3-carboxylic acid).

B.p. 140–2°/20 mm.

Perkin, *J. Chem. Soc.*, 1911, **99**, 738.

### $\Delta^1$ -Tetrahydro-*m*-toluic Acid (1-Methylcyclohexene-3-carboxylic acid)



$\text{C}_8\text{H}_{12}\text{O}_2$

MW, 140

*l.*

$[\alpha]_D - 49.7^\circ$  in AcOEt.

*Me ester*:  $\text{C}_9\text{H}_{14}\text{O}_2$ . MW, 154. B.p. 144–7°/100 mm.

*dl.*

B.p. 140–2°/20 mm., 123°/7 mm. Ox.  $\longrightarrow$  adipic acid + 4-aceto-*n*-valeric acid.

*Et ester*:  $C_{10}H_{16}O_2$ . MW, 168. B.p. 128°/60 mm.

Perkin, Tattersall, *J. Chem. Soc.*, 1907, **91**, 496.

Haworth, Perkin, *J. Chem. Soc.*, 1913, **103**, 2237.

Boorman, Linstead, *J. Chem. Soc.*, 1935, **264**.

**$\Delta^2$ -Tetrahydro-*m*-toluic Acid** (3-Methylcyclohexene-1-carboxylic acid).

M.p. 26°. B.p. 150°/11 mm., 130°/7 mm. Spar. sol.  $H_2O$ . Ox.  $\longrightarrow$  1-methyladipic acid.

See last reference above and also

Perkin, Tattersall, *J. Chem. Soc.*, 1905, **87**, 1095.

**$\Delta^3$ -Tetrahydro-*m*-toluic Acid** (5-Methylcyclohexene-1-carboxylic acid).

*d.*

M.p. 62–4°.  $[\alpha]_D + 40.1^\circ$  in AcOEt. Volatile in steam.

*Et ester*: b.p. 150–1°/100 mm.  $[\alpha]_D + 32.5^\circ$  in AcOEt.

*l.*

$[\alpha]_D - 35.8^\circ$  in AcOEt.

*dl.*

Plates from formic acid. M.p. 60°. B.p. 155–60°/25 mm. Very sol. most solvents. Ox.  $\longrightarrow$  2-methyladipic acid.

*Et ester*: b.p. 146–8°/100 mm., 105°/11 mm.  $D_4^{18}$  0.9762.  $n_D^{20}$  1.4695.

Auwers, *Ann.*, 1923, **432**, 98.

Perkin, Tattersall, *J. Chem. Soc.*, 1905, **87**, 1093.

Luff, Perkin, *J. Chem. Soc.*, 1910, **97**, 2151.

**$\Delta^4$ -Tetrahydro-*m*-toluic Acid** (5-Methylcyclohexene-3-carboxylic acid).

B.p. 143–6°/20 mm.

*Et ester*: b.p. 142–4°/100 mm.

Perkin, *J. Chem. Soc.*, 1910, **97**, 2146.

**$\Delta^5$ -Tetrahydro-*m*-toluic Acid** (6-Methylcyclohexene-4-carboxylic acid).

*d.*

B.p. 142–5°/20 mm.  $[\alpha]_D^{18} + 33^\circ$  in AcOEt.

*Et ester*: b.p. 140–1°/100 mm.  $[\alpha]_D^{18} + 30.5^\circ$  in AcOEt.

*l.*

B.p. 142°/20 mm.  $[\alpha]_D^{18} - 30.9^\circ$  in AcOEt.

*Et ester*: b.p. 140–2°/100 mm.  $[\alpha]_D^{18} - 27.4^\circ$  in AcOEt.

*dl.*

B.p. 177–80°/100 mm., 145°/20 mm.

*Et ester*: b.p. 141–3°/100 mm.

Perkin, *J. Chem. Soc.*, 1910, **97**, 2139.

**$\Delta^6$ -Tetrahydro-*m*-toluic Acid** (2-Methylcyclohexene-4-carboxylic acid).

*d.*

B.p. 138–40°/18 mm.  $[\alpha]_D + 108^\circ$  in AcOEt.

*l.*

$[\alpha]_D - 98.6^\circ$  in AcOEt.

*dl.*

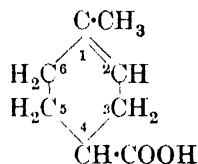
B.p. 184–6°/100 mm.

*Et ester*: b.p. 146°/100 mm.

Fisher, Perkin, *J. Chem. Soc.*, 1908, **93**, 1886.

Haworth, Perkin, *J. Chem. Soc.*, 1913, **103**, 2233.

**$\Delta^1$ -Tetrahydro-*p*-toluic Acid** (1-Methylcyclohexene-4-carboxylic acid)



$C_8H_{12}O_2$

MW, 140

*d.*

M.p. about 99°.  $[\alpha]_D + 55^\circ$ .

*Et ester*:  $C_{10}H_{16}O_2$ . MW, 168. B.p. 145–7°/100 mm.  $[\alpha]_D + 50^\circ$ .

*l.*

Cryst. from pet. ether. M.p. about 99°.  $[\alpha]_D - 58^\circ$ .

*Et ester*: b.p. 145–7°/100 mm.  $[\alpha]_D - 52^\circ$ .

*dl.*

Needles from  $H_2O$ . M.p. 99°. B.p. 140–5°/15 mm. Sol. AcOH. Spar. sol.  $H_2O$ . Conc.  $H_2SO_4 \longrightarrow p$ -toluic acid.

*Et ester*: b.p. 155–7°/100 mm., 105–8°/12 mm.

*Chloride*:  $C_8H_{11}OCl$ . MW, 158.5. B.p. 110–14°/40 mm., 90°/19 mm.

*Amide*:  $C_8H_{13}ON$ . MW, 139. Needles or leaflets from  $H_2O$ . M.p. 182°.

Lehmann, Paasche, *Ber.*, 1935, **68**, 1069.

Meldrum, Perkin, *J. Chem. Soc.*, 1908, **93**, 1424.

$\Delta^3$ -Tetrahydro-*p*-toluic Acid (4-Methylcyclohexene-1-carboxylic acid).

*d*-.  
 Prisms from  $H_2O$ . M.p.  $136-7^\circ$  ( $133^\circ$ ).  $[\alpha]_D + 100.1^\circ$  in  $AcOEt$ .

*Et ester*: b.p.  $154^\circ/100$  mm.  $[\alpha]_D + 86.5^\circ$ .  
 $D_{20}^{20} 0.9757$ .  $n_D^{20} 1.4688$ .

*l*-.  
 Prisms from  $AcOEt$ . M.p.  $133-4^\circ$ . Sol.  $AcOH$ ,  $AcOEt$ , pet. ether. Spar. sol. cold  $EtOH$ .  $[\alpha]_D^{17} - 100.8^\circ$  in  $AcOEt$ .

*Et ester*: b.p.  $154^\circ/100$  mm.  $[\alpha]_D^{18} - 83.5^\circ$ .

*dl*-.  
 Prismatic needles from  $AcOH.Aq$ . M.p.  $132-4^\circ$ . Very sol.  $EtOH$ ,  $Et_2O$ ,  $AcOH$ ,  $CHCl_3$ ,  $C_6H_6$ . Less sol. cold  $CS_2$ , ligroin.

*Et ester*: b.p.  $152-3^\circ/100$  mm.  $D_{16}^{15} 0.9792$ .  
 $n_a^{15} 1.4659$ .

*Amide*: leaflets from  $EtOH.Aq$ . M.p.  $148^\circ$ . Sol.  $EtOH$ . Spar. sol.  $H_2O$ .

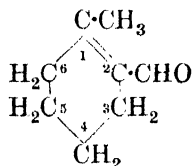
Einhorn, Willstätter, *Ann.*, 1894, **280**, 163.

Perkin, Pickles, *J. Chem. Soc.*, 1905, **87**, 645.

Kay, Perkin, *J. Chem. Soc.*, 1906, **89**, 844.

Chou, Perkin, *J. Chem. Soc.*, 1911, **99**, 534.

$\Delta^1$ -Tetrahydro-*o*-toluic Aldehyde (2-Methylcyclohexene-1-aldehyde)



$C_8H_{12}O$

MW, 124

Needles. M.p.  $98-101^\circ$ . B.p.  $75-6^\circ/12$  mm.

*Semicarbazone*: cryst. from  $MeOH$ . M.p.  $208-12^\circ$ . Insol.  $H_2O$ .

*p*-Nitrophenylhydrazone: cryst. from  $AcOH$ . M.p.  $160-3^\circ$ .

Bernhauer, Neubauer, *Biochem. Z.*, 1932, **251**, 173.

$\Delta^4$ -Tetrahydro-*o*-toluic Aldehyde (5-Methylcyclohexene-4-aldehyde).

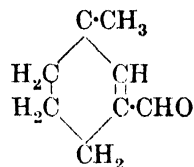
Liq. B.p.  $83^\circ/38$  mm.,  $75^\circ/22$  mm.

*Semicarbazone*: m.p.  $168^\circ$ .

Diels, Alder, *Ann.*, 1929, **470**, 85.

I.G., U.S.P., 1,944,731, (*Chem. Abstracts*, 1934, **28**, 2016).

$\Delta^2$ -Tetrahydro-*m*-toluic Aldehyde (3-Methylcyclohexene-1-aldehyde)



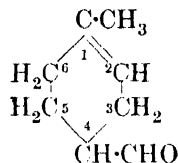
$C_8H_{12}O$

MW, 124

*Semicarbazone*: m.p.  $206-7^\circ$ . Spar. sol. most solvents.

Wallach, *Ann.*, 1906, **347**, 344.

$\Delta^1$ -Tetrahydro-*p*-toluic Aldehyde (1-Methylcyclohexene-4-aldehyde)



$C_8H_{12}O$

MW, 124

B.p.  $63-4^\circ/10$  mm.

*Semicarbazone*: m.p.  $146^\circ$ .

Diels, Alder, *Ann.*, 1929, **470**, 87.

See also Lehmann, Paasche, *Ber.*, 1935, **68**, 1068.

$\Delta^3$ -Tetrahydro-*p*-toluic Aldehyde (4-Methylcyclohexene-1-aldehyde).

*Semicarbazone*: m.p.  $192-4^\circ$ .

Wallach, Evans, *Ann.*, 1906, **347**, 346.

**Tetrahydrotoluquinoline.**

See 6-, 7-, and 8-Methyltetrahydroquinolines.

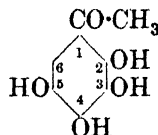
**Tetrahydrotolylacetic Acid.**

See Methylcyclohexenylacetic Acid.

**Tetrahydrotubanol.**

See 2-Isoamylresorcinol.

2 : 3 : 4 : 5-Tetrahydroxyacetophenone



$C_8H_8O_5$

MW, 184

3 : 4-Di-Me ether:  $C_{10}H_{12}O_5$ . MW, 212.

Yellow needles from  $Et_2O$ . M.p.  $119-21^\circ$ .

Sol.  $EtOH$ ,  $CHCl_3$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  yellow sol.

Bargellini, *Gazz. chim. ital.*, 1916, **46**, i, 253.

**2 : 3 : 4 : 6-Tetrahydroxyacetophenone.**

Pale yellow needles from EtOH or AcOH.Aq. M.p. 204–5°.

3 : 4-Di-Me ether : needles from EtOH.Aq. M.p. 166–8° (162–3°). Conc. H<sub>2</sub>SO<sub>4</sub> → orange-yellow sol.

2 : 3 : 4-Tri-Me ether : C<sub>11</sub>H<sub>14</sub>O<sub>5</sub>. MW, 226. Needles from EtOH. M.p. 164–5°.

3 : 4 : 6-Tri-Me ether : cubes from EtOH. M.p. 112–13° (125–6°). Acetyl : m.p. 106°. Benzoyl : m.p. 120–2°.

Tetra-Me ether : C<sub>12</sub>H<sub>16</sub>O<sub>5</sub>. MW, 240. Needles from EtOH.Aq. M.p. 53–4° (43–5°). B.p. 310°. Oxime : m.p. 103–5°. Semicarbazone : m.p. 128–30°.

Phenylhydrazone : red prismatic needles. M.p. 248–51° decomp.

Bargellini, Bini, *Gazz. chim. ital.*, 1911, 41, ii, 18.

Bargellini, Zoras, *Gazz. chim. ital.*, 1934, 64, 192.

Bargellini, *Gazz. chim. ital.*, 1919, 49, ii, 47.

Nierenstein, *J. Chem. Soc.*, 1917, 111, 6.

**Tetrahydroxyacetophenone.**

See also Trihydroxyphenacyl Alcohol.

**Tetrahydroxyadipic Acid.**

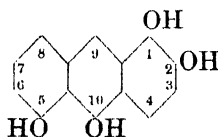
See Mucic Acid, Allomucic Acid, and Saccharic Acid.

**Tetrahydroxy-1-aminocaproic Acid.**

See Glucosaminic Acid.

**1 : 2 : 5 : 10-Tetrahydroxyanthracene**

(1 : 5 : 6-Trihydroxyanthrone, 1 : 5 : 6-trihydroxyanthranol)



C<sub>14</sub>H<sub>10</sub>O<sub>4</sub>

MW, 242

Cryst. from EtOH.Aq. M.p. 258°.

Mono-Me ether : C<sub>15</sub>H<sub>12</sub>O<sub>4</sub>. MW, 256. Cryst. from EtOH. M.p. 140°.

Graebe, Thode, *Ann.*, 1906, 349, 218.

**1 : 2 : 6 : 10-Tetrahydroxyanthracene**

(2 : 5 : 6-Trihydroxyanthrone, 2 : 5 : 6-trihydroxyanthranol).

Yellow cryst. M.p. 258°. NaOH → red-dish-yellow sol. Conc. H<sub>2</sub>SO<sub>4</sub> → brownish-yellow sol.

Tri-Me ether : C<sub>17</sub>H<sub>16</sub>O<sub>4</sub>. MW, 284. Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 169–70°. Sol. hot C<sub>6</sub>H<sub>6</sub>. Mod. sol. hot EtOH, AcOH.

Tetra-acetyl : m.p. 250–60°.

Bayer, D.R.P., 117,923, (*Chem. Zentr.*, 1901, I, 600).

Graebe, Thode, *Ann.*, 1906, 349, 214.

Bistrzycki, Yssel de Schepper, *Ber.*, 1898, 31, 2799.

**1 : 2 : 7 : 10-Tetrahydroxyanthracene**

(3 : 4 : 6-Trihydroxyanthrone, 3 : 4 : 6-trihydroxyanthranol).

Yellow needles.

1 : 2 : 7-Tri-Me ether : needles from EtOH. M.p. 149°. Acetyl : needles. M.p. 127°.

Tetra-acetyl : needles from EtOH. M.p. 167°.

MacMaster, Perkin, *J. Chem. Soc.*, 1927, 1309.

Graebe, Bernhard, *Ann.*, 1906, 349, 227.

Liebermann, *Ber.*, 1888, 21, 443.

**1 : 2 : 9 : 10-Tetrahydroxyanthracene**

(1 : 2 : 10-Trihydroxyanthrone, 1 : 2 : 10-trihydroxyanthranol, 1 : 2-dihydroxyanthrahydroquinone, leuco-alizarin).

Brown leaflets from AcOH. M.p. 150°. Very sol. EtOH with yellow col. Conc. H<sub>2</sub>SO<sub>4</sub> → brown col. → red on standing. NaOH → red sol.

Grandmougin, *J. prakt. Chem.*, 1907, 76, 141.

**1 : 3 : 9 : 10-Tetrahydroxyanthracene**

(Dihydropurpuroxanthin, leuco-purpuroxanthin, 1 : 3 : 10-trihydroxyanthrone, 1 : 3 : 10-trihydroxyanthranol, 1 : 3-dihydroxyanthrahydroquinone).

Pale yellow needles. Sol. Et<sub>2</sub>O. NaOH → brown sol. Oxidises in air to purpuroxanthin.

Rosenstiehl, *Ann. chim. phys.*, 1879, 18, 230.

**1 : 4 : 9 : 10-Tetrahydroxyanthracene** (Dihydroquinizarin, leuco-quinizarin, 1 : 4 : 10-trihydroxyanthrone, 1 : 4 : 10-trihydroxyanthranol, 1 : 4-dihydroxyanthrahydroquinone).

Yellow needles. M.p. 156° (131–6°). Alkalis → red sols. Sol. EtOH, AcOH with yellow col. and weak blue fluor. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

Tetra-acetyl : pale yellow cryst. from AcOH. M.p. 240–2°.

M.L.B., D.R.P., 207,668, (*Chem. Zentr.*, 1909, I, 1287).

Zahn, Ochwat, *Ann.*, 1928, 462, 72.

Meyer, Sander, *Ann.*, 1920, 420, 123.

**2 : 3 : 6 : 7-Tetrahydroxyanthracene.**

*Tetra-Me ether*:  $C_{18}H_{18}O_4$ . MW, 298.  
Cryst. from EtOH. M.p. 173°. Most sols.  
show blue fluor.

Robinson, *J. Chem. Soc.*, 1915, 107, 272.

**2 : 3 : 9 : 10-Tetrahydroxyanthracene** (*Di-hydrokystazarin*, 2 : 3 : 9-trihydroxyanthrone, 2 : 3 : 9-trihydroxyanthranol, *leuco-kystazarin*).

*Tetra-acetyl*: needles from EtOH. M.p. 217–19°.

Schöller, *Ber.*, 1889, 22, 684.

**2 : 6 : 9 : 10-Tetrahydroxyanthracene** (*Di-hydroanthraflavic acid*, 2 : 6 : 10-trihydroxyanthrone, 2 : 6 : 10-trihydroxyanthranol, *leuco-anthraflavic acid*).

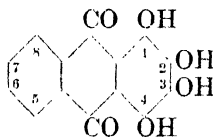
*Tetra-acetyl*: needles. M.p. 274°.

Liebermann, *Ber.*, 1888, 21, 1173.

**2 : 7 : 9 : 10-Tetrahydroxyanthracene** (*Di-hydroisoanthraflavic acid*, 2 : 7 : 10-trihydroxyanthrone, 2 : 7 : 10-trihydroxyanthranol, *leuco-iso-anthraflavic acid*).

*Tetra-acetyl*: needles. M.p. 235–40°.

Liebermann, *Ber.*, 1888, 21, 1173.

**1 : 2 : 3 : 4-Tetrahydroxyanthraquinone**

$C_{14}H_8O_6$

MW, 272

Green needles. Conc.  $H_2SO_4 \rightarrow$  red sol.  
KOH  $\rightarrow$  red sol.

*Tetra-acetyl*: yellow needles. M.p. 205°.  
Sol. boiling KOH with violet-red col.

Slama, *Chem. Zentr.*, 1899, II, 966.

Bayer, D.R.P., 125,579, (*Chem. Zentr.*, 1901, II, 1188).

**1 : 2 : 3 : 5-Tetrahydroxyanthraquinone** ( $\alpha$ -*Hydroxyanthragallol*).

Yellow needles from EtOH. Does not melt below 350°. Sol. EtOH,  $Me_2CO$ , AcOH. Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  violet sol.

*Tetra-acetyl*: needles from AcOH. M.p. 207–9°.

Liebermann, Kostanecki, *Ann.*, 1888, 244, 360; 1887, 240, 270.

**1 : 2 : 3 : 7-Tetrahydroxyanthraquinone** ( $\beta$ -*Hydroxyanthragallol*).

Red needles from EtOH. M.p. 380°. Sol. EtOH,  $Me_2CO$ , AcOH. Spar. sol.  $H_2O$ ,  $Et_2O$ .

Insol.  $CHCl_3$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  brownish-yellow sol. Sublimes slowly with decomp.

*Tetra-acetyl*: yellow plates from AcOH. M.p. 189°. Sol. EtOH,  $CHCl_3$ , AcOH.

Liebermann, Kostanecki, Noah, *Ann.*, 1888, 244, 360; 1887, 240, 271.

**1 : 2 : 4 : 6-Tetrahydroxyanthraquinone** (*Hydroxyflavopurpurin*).

Cryst. Sol. EtOH, Py. Less sol. AcOH.  
Conc.  $H_2SO_4 \rightarrow$  bluish-red sol.

*Tetra-acetyl*: m.p. 202°.

Dimroth, Fick, *Ann.*, 1916, 411, 324.

Bayer, D.R.P., 67,061.

**1 : 2 : 4 : 7-Tetrahydroxyanthraquinone** (*Hydroxyanthrapurpurin*).

Cryst. from EtOH, Py, or AcOH. Sol. EtOH, AcOH. NaOH  $\rightarrow$  bluish-red sol. Conc.  $H_2SO_4 \rightarrow$  cherry-red sol.

2 : 7-Diacetyl: orange needles from AcOH. M.p. 224–5°. Sol.  $AcOEt$ .

*Tetra-acetyl*: m.p. 214–5°.

Dimroth, Friedemann, Kammerer, *Ber.*, 1920, 53, 483.

Dimroth, Fick, *Ann.*, 1916, 411, 328.

Bayer, D.R.P., 67,061.

**1 : 2 : 5 : 6-Tetrahydroxyanthraquinone.**  
See Rufiopin.**1 : 2 : 5 : 8-Tetrahydroxyanthraquinone.**  
See Quinalizarin.

**1 : 2 : 6 : 7-Tetrahydroxyanthraquinone.**  
Orange needles from  $PhNO_2$ . Does not melt below 330°. Mod. sol.  $PhNO_2$ . Spar. sol. EtOH,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  violet sol. NaOH  $\rightarrow$  violet sol.

1 : 2 : 6-Tri-Me ether:  $C_{17}H_{14}O_6$ . MW, 314.  
Yellow needles from AcOH. M.p. 269–70°.

*Tetra-Me ether*:  $C_{18}H_{16}O_6$ . MW, 328. Yellow needles from AcOH. M.p. 244–5° (239°).

*Tetra-acetyl*: cryst. from AcOH. M.p. 239–41°.

Bistrzycki, Krauer, *Helv. Chim. Acta*, 1923, 6, 768.

Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, 47, 2017.

**1 : 2 : 7 : 8-Tetrahydroxyanthraquinone.**  
Red prisms from AcOH. M.p. 315° (292°)

decomp. Sol. Py. Spar. sol. EtOH, AcOH,  $PhNO_2$ , toluene. NaOH  $\rightarrow$  blue sol. Conc.  $H_2SO_4 \rightarrow$  violet-red sol. Sublimes.

*Diacetyl deriv.*: m.p. 242°.

*Tetra-acetyl*: yellow needles. M.p. 238–40° decomp.

Bayer, D.R.P., 103,988, (*Chem. Zentr.*, 1899, II, 922).

Heller, *Z. angew. Chem.*, 1929, 42, 170.

**1 : 3 : 5 : 7-Tetrahydroxyanthraquinone.**  
See Anthrachrysazin.

**1 : 4 : 5 : 8-Tetrahydroxyanthraquinone.**  
Brown needles from  $C_6H_6$ -ligroin. Does not melt below 300° (246°). Insol.  $H_2O$ . NaOH  $\rightarrow$  blue sol. Conc.  $H_2SO_4 \rightarrow$  greenish-blue sol. with red. fluor.

*Tetra-Me ether*: orange leaflets from AcOH. M.p. 317°. Sol. boiling AcOH, xylene,  $PhNO_2$ . Spar. sol. EtOH,  $C_6H_6$ , hot  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  greenish-blue sol.

*Tetra-acetyl*: pale yellow needles from AcOH. Decomp. above 258°. Conc.  $H_2SO_4 \rightarrow$  blue col.

Fischer, Ziegler, *J. prakt. Chem.*, 1912, 86, 300.

Frey, *Ber.*, 1912, 45, 1361.

Bayer, D.R.P., 143,804, (*Chem. Zentr.*, 1903, II, 475).

S.C.I., Bâle, D.R.P., 533,340, (*Chem. Abstracts*, 1932, 26, 480).

Marshall, *J. Chem. Soc.*, 1937, 255.

**2 : 3 : 6 : 7-Tetrahydroxyanthraquinone.**  
Orange-yellow. Turns brown at 360°. Sol. alkalis with greenish-yellow col.

*Tetra-Me ether*: yellow needles from AcOH or  $PhNO_2$ . M.p. 346° (344°). Conc.  $H_2SO_4 \rightarrow$  green sol. Sublimes.

**2 : 6-Di-Me-3 : 7-di-Et ether**:  $C_{20}H_{20}O_6$ . MW, 356. Yellow needles from AcOH. M.p. 288°. Conc.  $H_2SO_4 \rightarrow$  green sol.

**2 : 3 : 6-Tri-Me-7-Et ether**:  $C_{19}H_{18}O_6$ . MW, 342. Yellow needles from AcOH. M.p. 290°. Conc.  $H_2SO_4 \rightarrow$  green sol.

*Tetra-acetyl*: yellow cryst. from AcOH. M.p. 300° decomp.

Vanzetti, Dreyfus, *Gazz. chim. ital.*, 1934, 64, 392.

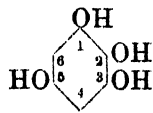
Vanzetti, Oliverio, *Gazz. chim. ital.*, 1930, 60, 620.

Haworth, Mavin, *J. Chem. Soc.*, 1931, 1365.

**1 : 2 : 3 : 4-Tetrahydroxybenzene.**

See Apionol.

**1 : 2 : 3 : 5-Tetrahydroxybenzene**



$C_6H_4O_4$

MW, 142

Needles from  $H_2O$ . M.p. 165°. Sol.  $H_2O$ , EtOH, AcOEt. Insol.  $CHCl_3$ ,  $C_6H_6$ . Decomp. easily in air.  $FeCl_3 \rightarrow$  red col.

*2-Me ether*: see Iretol.

**1 : 3-Di-Me ether**:  $C_8H_{10}O_4$ . MW, 170. Needles. M.p. 159°. Sol. EtOH,  $Et_2O$ . Sol. alkalis with green col. Decomp. in moist air. *2-Et ether*:  $C_{10}H_{14}O_4$ . MW, 198. Yellowish needles from ligroin. M.p. 119°. Sol. most org. solvents.  $FeCl_3 \rightarrow$  green col. *Diacetyl*: needles from boiling  $H_2O$ . M.p. 128°. Sol. EtOH. Spar. sol.  $H_2O$ .

**1 : 2 : 3-Tri-Me ether**: see Antiarol.

*Tetra-Me ether*: see under Antiarol.

*2-Et ether*:  $C_8H_{10}O_4$ . MW, 170. Needles. M.p. 220°. *Triacetyl*: cryst. from  $C_6H_6$ -ligroin. M.p. 74°. B.p. 232°/17 mm.

**1 : 2 : 3-Tri-Et ether**:  $C_{12}H_{18}O_4$ . MW, 226. Needles from  $H_2O$ . M.p. 105°. Sol. EtOH,  $Et_2O$ .

Oettinger, *Monatsh.*, 1895, 16, 256.

Will, *Ber.*, 1888, 21, 610.

Kohner, *Monatsh.*, 1899, 20, 938.

Nierenstein, *J. Chem. Soc.*, 1917, 111, 5.

**1 : 2 : 4 : 5-Tetrahydroxybenzene.**

Leaflets from AcOH. M.p. 215–20°. Very sol.  $H_2O$ , EtOH,  $Et_2O$ . Less sol. AcOH. Aq. sol. turns brown in air.

*2-Me ether*: *triacetyl*, cryst. from MeOH or AcOH. M.p. 142°.

**1 : 4-Di-Me ether**: needles. M.p. 170° (166°). Sol. EtOH, warm  $CHCl_3$ .

*Tetra-Me ether*:  $C_{10}H_{14}O_4$ . MW, 198. Needles from  $H_2O$ . M.p. 103°. Sol. EtOH,  $C_6H_6$ . Spar. sol. cold  $H_2O$ .

**1 : 4-Di-Et ether**:  $C_{10}H_{14}O_4$ . MW, 198. Needles from hot  $H_2O$ . M.p. 138°. *Diacetyl*: leaflets. M.p. 148°.

*Tetra-Et ether*:  $C_{14}H_{22}O_4$ . MW, 254. Leaflets from EtOH.Aq. M.p. 143°. Sublimes. Volatile in steam.

**1 : 4-Dipropyl ether**:  $C_{12}H_{18}O_4$ . MW, 226. Needles from ligroin. M.p. 95°. Very sol. most solvents. Conc.  $H_2SO_4 \rightarrow$  greenish sol.

*Tetra-acetyl*: leaflets from AcOH. M.p. 226–7° (217°).

Jackson, Beggs, *J. Am. Chem. Soc.*, 1914, 36, 1216.

Nietzki, Schmidt, *Ber.*, 1888, 21, 2377.

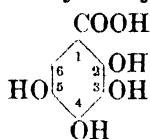
Schüler, *Arch. Pharm.*, 1907, 245, 281.

Nietzki, Reichberg, *Ber.*, 1890, 23, 1214.

Erdtmann, *Chem. Abstracts*, 1934, 28, 1337.



## 2 : 3 : 4 : 5-Tetrahydroxybenzoic Acid

 $C_7H_6O_6$ 

MW, 186

2 : 5-Di-Me ether :  $C_9H_{10}O_6$  MW, 214. Cryst. from AcOEt. M.p. 147° 8°. Sol.  $H_2O$ , EtOH, Et<sub>2</sub>O, AcOEt. Insol. ligroin.  $FeCl_3 \rightarrow$  blue col. Di-Et ether :  $C_{13}H_{18}O_6$  MW, 270. Pale yellow cryst. from pet. ether. M.p. 83°. B.p. 95°/0.2 mm.

3 : 5-Di-Me ether : pale yellow cryst. M.p. 165° decomp. Sol. EtOH,  $C_6H_6$ .  $FeCl_3 \rightarrow$  brown col. Diacetyl : cryst. Decomp. at 112° (162°). Sol. EtOH. Insol.  $H_2O$ .

3 : 4 : 5-Tri-Me ether :  $C_{10}H_{12}O_6$  MW, 228. Cryst. M.p. 191° decomp. Sol. EtOH, Et<sub>2</sub>O,  $C_6H_6$ . Spar. sol.  $H_2O$ , ligroin. Me ester :  $C_{11}H_{14}O_6$  MW, 242. Cryst. M.p. 85°. Sol. EtOH, Et<sub>2</sub>O. Insol.  $H_2O$ .

Tetra-Me ether :  $C_{11}H_{14}O_6$  MW, 242. Needles from EtOH.Aq. M.p. 87.5°. Sol. EtOH, boiling  $H_2O$ ,  $Me_2CO$ , AcOEt,  $C_6H_6$ .

Bartolotti, *Gazz. chim. ital.*, 1892, **22**, i, 562.

Hamburg, *Monatsh.*, 1898, **19**, 603.

Wesseley, Demmer, *Ber.*, 1928, **61**, 1281.

Bogert, Plant, *J. Am. Chem. Soc.*, 1915, **37**, 2733.

## 2 : 3 : 4 : 6-Tetrahydroxybenzoic Acid.

Needles from  $H_2O$ . M.p. 308–10° decomp.

Tetra-Me ether : needles from EtOH. M.p. 184–6° (149–50°). Me ester :  $C_{12}H_{16}O_6$  MW, 256. Needles from  $C_6H_6$ . M.p. 134–6°. Chloride :  $C_{11}H_{13}O_5Cl$  MW, 260.5. Needles from pet. ether. M.p. 104°. Nitrile :  $C_{11}H_{13}O_4N$  MW, 223. Needles from EtOH. M.p. 114°.

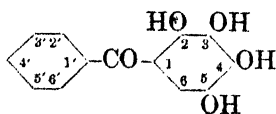
Tetra-acetyl : needles from MeOH. M.p. 274–6°.

Tetrabenzoyle : prismatic needles from EtOH. M.p. 248–9°.

Nierenstein, *J. Chem. Soc.*, 1917, **111**, 5.

Bargellini, Madesani, *Gazz. chim. ital.*, 1931, **61**, 684.

## 2 : 3 : 4 : 5-Tetrahydroxybenzophenone

 $C_{13}H_{10}O_5$ 

MW, 246

3 : 4-Di-Me ether :  $C_{15}H_{14}O_5$  MW, 274. Yellow prisms from  $H_2O$ . M.p. 140–2°. Sol. EtOH, AcOH,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  yellow sol.

Bargellini, *Gazz. chim. ital.*, 1916, **46**, i, 254.

## 2 : 3 : 4 : 6-Tetrahydroxybenzophenone.

Tri-Me ether :  $C_{16}H_{16}O_5$  MW, 288. Needles from ligroin. M.p. 87–9°. Sol. EtOH,  $CHCl_3$ ,  $Me_2CO$ ,  $C_6H_6$ . Insol.  $H_2O$ . Alc.  $FeCl_3 \rightarrow$  reddish-brown col. Conc.  $H_2SO_4 \rightarrow$  yellow sol.  $\rightarrow$  green  $\rightarrow$  dark violet on warming. Acetyl : needles from EtOH.Aq. M.p. 130–2°. Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ .

Tetra-Me ether :  $C_{17}H_{18}O_5$  MW, 302. Needles from ligroin. M.p. 125–6°. Sol. EtOH,  $Me_2CO$ ,  $C_6H_6$ . Insol.  $H_2O$ . Conc.  $H_2SO_4 \rightarrow$  yellow sol.  $\rightarrow$  dark violet on warming.

Bargellini, *Gazz. chim. ital.*, 1915, **45**, i, 89.

## 2 : 3 : 4 : 2'-Tetrahydroxybenzophenone.

Yellow plates +  $H_2O$  from  $H_2O$ . M.p. 100°, anhyd. 149°. Sol. EtOH, AcOH, Et<sub>2</sub>O. Spar. sol.  $C_6H_6$ , ligroin. Sublimes.

Tetra-acetyl : cryst. from EtOH or AcOH. M.p. 118°.

Badische, D.R.P., 49,149.

Graebe, Eichengrün, *Ann.*, 1892, **269**, 307.

Atkinson, Heilbron, *J. Chem. Soc.*, 1926, 2690.

## 2 : 3 : 4 : 3'-Tetrahydroxybenzophenone.

Yellow needles. M.p. 133°.

See first reference above.

## 2 : 3 : 4 : 4'-Tetrahydroxybenzophenone.

Yellowish needles. M.p. above 200°.

Badische, D.R.P., 49,149.

## 2 : 4 : 5 : 4'-Tetrahydroxybenzophenone.

4 : 5 : 4'-Tri-Me ether : yellow needles from EtOH.Aq. M.p. 127–8°. Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Alc.  $FeCl_3 \rightarrow$  yellowish-green col.

Tetra-Me ether : yellow cryst. powder. M.p. 122–4°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  orange-yellow sol. Phenylhydrazones : m.p. 173–4°.

Bargellini, Martegiani, *Atti accad. Lincei*, 1911, **20**, II, 188.

## 2 : 4 : 6 : 2'-Tetrahydroxybenzophenone.

Golden-yellow leaflets from EtOH. Blackens on heating. Very spar. sol. EtOH. Insol.

H<sub>2</sub>O, most org. solvents. Has paralytic action on heart in large doses.

Karrer, *Helv. Chim. Acta*, 1921, **4**, 992.

**2 : 4 : 6 : 3'-Tetrahydroxybenzophenone.**

Pale yellow leaflets from H<sub>2</sub>O. Decomp. at 246°. Sol. EtOH, hot H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O. Alc. FeCl<sub>3</sub> → purple col. Alkalis → orange sols. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

Nishikawa, Robinson, *J. Chem. Soc.*, 1922, **121**, 842.

**2 : 4 : 6 : 4'-Tetrahydroxybenzophenone.**

Pale brown prisms from H<sub>2</sub>O. M.p. 210°. Alc. FeCl<sub>3</sub> → brown col. Alkalis → yellow sols.

*Tetra-Me ether*: prisms from EtOH. M.p. 146°.

4'-Carboethoxyl: yellow needles from EtOH.Aq. M.p. 172°.

Nishikawa, Robinson, *J. Chem. Soc.*, 1922, **121**, 843.

Kostanecki, Tambor, *Ber.*, 1906, **39**, 4024.

**2 : 3 : 2' : 3'-Tetrahydroxybenzophenone.**

*Tetra-Me ether*: cryst. M.p. 145°.

Staudinger, Schlenker, Goldstein, *Helv. Chim. Acta*, 1921, **4**, 341.

**2 : 4 : 2' : 4'-Tetrahydroxybenzophenone.**

Yellow needles + 1½ H<sub>2</sub>O from H<sub>2</sub>O. M.p. 193-5°. Sol. MeOH, EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Mod. sol. hot H<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. FeCl<sub>3</sub> → dark brown col.

*Tetra-Me ether*: yellowish leaflets from EtOH.Aq. M.p. 130°. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

Tambor, *Ber.*, 1910, **43**, 1889.

Meyer, Conzetti, *Ber.*, 1897, **30**, 971.

Shoosmith, Haldane, *J. Chem. Soc.*, 1924, **125**, 113.

**2 : 4 : 2' : 6'-Tetrahydroxybenzophenone.**

Cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. about 200°. Sol. hot H<sub>2</sub>O.

Graebe, *Ann.*, 1889, **254**, 302.

**2 : 4 : 3' : 4'-Tetrahydroxybenzophenone.**

Needles + 2H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 202°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOH. Less sol. H<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. ligroin. Aq. sol. shows green fluor.

3'-*Me ether*: C<sub>14</sub>H<sub>12</sub>O<sub>5</sub>. MW, 260. Yellowish needles from H<sub>2</sub>O. M.p. 210°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. FeCl<sub>3</sub> → intense red-dish-brown col.

*Tetra-Me ether*: prisms from EtOH. M.p. 126° (107°). Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

M.L.B., D.R.P., 72,446.

Hoesch, v. Zarzecki, *Ber.*, 1917, **50**, 465.

Komarowski, Kostanecki, *Ber.*, 1894, **27**, 2000.

Mitter, Paul, *J. Indian Chem. Soc.*, 1931, **8**, 274.

**2 : 4 : 3' : 5'-Tetrahydroxybenzophenone.**

*Tetra-Me ether*: needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 73-4°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. AcOH. Insol. pet. ether. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 407.

**2 : 5 : 2' : 5'-Tetrahydroxybenzophenone.**

*Tetra-Me ether*: yellowish cryst. from EtOH. M.p. 109°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Less sol. EtOH, Et<sub>2</sub>O, AcOH. *Oxime*: cryst. M.p. 134-5°. *Phenylhydrazone*: m.p. 170°.

Kauffmann, Grombach, *Ann.*, 1906, **344**, 74.

Kauffmann, Fritz, *Ber.*, 1908, **41**, 4425.

Staudinger, Schlenker, Goldstein, *Helv. Chim. Acta*, 1921, **4**, 341.

**2 : 5 : 2' : 6'-Tetrahydroxybenzophenone.**

Yellow needles from H<sub>2</sub>O. M.p. 200-2° decomp.

*Tetra-Et ether*: C<sub>21</sub>H<sub>26</sub>O<sub>5</sub>. MW, 358. Leaflets or needles from EtOH. M.p. 93-5°. Spar. sol. EtOH.

*Tetra-acetyl*: leaflets from EtOH. M.p. 118-19°.

Baeyer, *Ann.*, 1870, **155**, 259.

Herzig, *Monatsh.*, 1892, **13**, 412.

**2 : 5 : 3' : 4'-Tetrahydroxybenzophenone.**

*Tetra-Me ether*: prisms from EtOH.Aq. M.p. 101-2°. Conc. H<sub>2</sub>SO<sub>4</sub> → orange sol.

König, Kostanecki, *Ber.*, 1906, **39**, 4030.

**2 : 6 : 2' : 6'-Tetrahydroxybenzophenone.**

*Tetra-Me ether*: plates from C<sub>6</sub>H<sub>6</sub>. M.p. 204°. Very sol. CHCl<sub>3</sub>. Mod. sol. EtOH, AcOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Baeyer, *Ann.*, 1910, **372**, 130.

**3 : 4 : 3' : 4'-Tetrahydroxybenzophenone.**

Cryst. from H<sub>2</sub>O. M.p. 227-8°.

*Tetra-Me ether*: prisms from EtOH. M.p. 145°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether. *Oxime*: cryst. from EtOH. M.p. 145°.

M.L.B., D.R.P., 72,446.

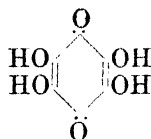
Kostanecki, Tambor, *Ber.*, 1906, **39**, 4027.

Perkin, Weizmann, *J. Chem. Soc.*, 1906, **89**, 1661.

**2 : 4 : 3' : 5'-Tetrahydroxybenzophenone.**

*Tetra-Me ether*: needles from  $C_6H_6$ . M.p. 114–15°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , hot  $C_6H_6$ . Spar. sol. AcOH. Insol. ligroin. Conc.  $H_2SO_4$  → yellow sol.

Mauthner, *J. prakt. Chem.*, 1913, **87**, 407.

**Tetrahydroxy-p-benzoquinone**

$C_6H_4O_6$  MW, 172

Bluish-black cryst. Very sol. EtOH, hot  $H_2O$ . Spar. sol.  $Et_2O$ , cold  $H_2O$ . Strong dibasic acid.

*Di-Na salt*: bluish-black cryst. with metallic green lustre.

*Diphenyl ether*:  $C_{18}H_{12}O_6$ . MW, 324. Reddish-brown plates from EtOH. M.p. 276°. Sol. AcOH. Spar. sol. EtOH, hot  $CHCl_3$ . Insol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ , ligroin. *Di-Me ether*:  $C_{20}H_{16}O_6$ . MW, 352. Golden-yellow needles from  $C_6H_6$ -EtOH. M.p. 171°. Sol.  $CHCl_3$ . Mod. sol. EtOH,  $C_6H_6$ , AcOH. Spar. sol.  $Et_2O$ ,  $CS_2$ . Insol. ligroin. *Di-Et ether*:  $C_{22}H_{20}O_6$ . MW, 380. Orange-yellow needles from EtOH. M.p. 128°. Sol. EtOH,  $CHCl_3$ . Spar. sol.  $Et_2O$ , AcOH.

*Tetraphenyl ether*:  $C_{30}H_{20}O_6$ . MW, 476. Red prisms from  $C_6H_6$ . M.p. 229–30°. Spar. sol.  $CS_2$ , boiling AcOH. Very spar. sol. warm  $Me_2CO$ . Insol. EtOH,  $Et_2O$ , ligroin.

*Diacetyl*: yellow leaflets. M.p. 205°. Mod. sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

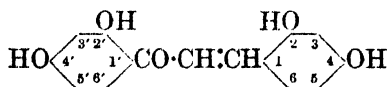
Jackson, Grindley, *Am. Chem. J.*, 1895, **17**, 647.

M.L.B., D.R.P., 368,741, (*Chem. Abstracts*, 1924, **18**, 991).

Maquenne, *Ann. chim. phys.*, 1887, **12**, 112.

**1 : 2 : 3 : 4-Tetrahydroxybutane.**

See Erythritol.

**2 : 4 : 2' : 4'-Tetrahydroxychalkone (2 : 4-Dihydroxyphenyl 2 : 4-dihydroxystyryl ketone)**

$C_{15}H_{12}O_5$  MW, 272

2 : 4 : 4'-*Tri-Me ether*:  $C_{18}H_{18}O_5$ . MW, 314. Yellow needles from EtOH. M.p. 157°. Conc.  $H_2SO_4$  → red sol. *Acetyl*: Pale yellow cryst. from EtOH. M.p. 110–12°.

4 : 2' : 4'-*Tri-Me ether*: greenish-yellow needles from  $Me_2CO$ . M.p. 156°.

*Tetra-Me ether*:  $C_{19}H_{20}O_5$ . MW, 328. Yellowish needles from EtOH. M.p. 128°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ , ligroin.

Bhalla, Rây, *J. Chem. Soc.*, 1933, 290.

Kostanecki, Lampe, Triulzi, *Ber.*, 1906, **39**, 92.

Kauffmann, Kieser, *Ber.*, 1913, **46**, 3797.

**2 : 4 : 2' : 5'-Tetrahydroxychalkone (2 : 5-Dihydroxyphenyl 2 : 4-dihydroxystyryl ketone).**

2 : 4 : 5'-*Tri-Me ether*: orange-yellow needles from EtOH. M.p. 118°. Conc.  $H_2SO_4$  → red sol. *Acetyl*: yellowish needles from EtOH. M.p. 87°.

*Tetra-Me ether*: cryst. from EtOH.Aq. M.p. 112°.

Kauffmann, Kieser, *Ber.*, 1913, **46**, 3798.

Bonifazi, Kostanecki, Tambor, *Ber.*, 1906, **39**, 88.

**3 : 4 : 2' : 4'-Tetrahydroxychalkone.**

See Butein.

**3 : 4 : 2' : 5'-Tetrahydroxychalkone (2 : 5-Dihydroxyphenyl 3 : 4-dihydroxystyryl ketone).**

3 : 5'-*Di-Me ether*:  $C_{17}H_{16}O_5$ . MW, 300. Orange-red leaflets from EtOH. M.p. 122–3°. Conc.  $H_2SO_4$  → yellowish-red sol.

Milobedzka, Kostanecki, Lampe, *Ber.*, 1910, **43**, 2164.

**3 : 4 : 3' : 4'-Tetrahydroxychalkone (3 : 4-Dihydroxyphenyl 3 : 4-dihydroxystyryl ketone).**

*Tetra-Me ether*: yellow prisms from EtOH. M.p. 116° (110°). Conc.  $H_2SO_4$  + AcOH → dark red sol.

Kauffmann, Kieser, *Ber.*, 1913, **46**, 3798.

Perkin, Rây, Robinson, *J. Chem. Soc.*, 1926, 951.

**3 : 5 : 3' : 4'-Tetrahydroxychalkone (3 : 4-Dihydroxyphenyl 3 : 5-dihydroxystyryl ketone).**

*Tetra-Me ether*: yellow needles from EtOH.Aq. M.p. 103–4°. Sol. EtOH,  $C_6H_6$ , warm ligroin. Insol. pet. ether. Conc.  $H_2SO_4$  → blood-red sol.

Mauthner, *J. prakt. Chem.*, 1920, **100**, 182.

**2 : 2' : 3' : 4' - Tetrahydroxychalkone (2 : 3 : 4-Trihydroxyphenyl 2-hydroxystyryl ketone).**

Yellow needles from 75% MeOH. M.p. 224–5° decomp. Very sol. EtOH,  $Me_2CO$ . Spar. sol. boiling xylene. Insol.  $H_2O$ ,  $CHCl_3$ .

$C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  deep brown sol.  
Alkalis  $\rightarrow$  deep brown sols.

2 : 3' : 4' - *Tri-Me ether*: pale yellow needles from EtOH. M.p. 105°. Conc.  $H_2SO_4 \rightarrow$  orange-red sol. *Acetyl*: needles from EtOH. M.p. 88°.

Ellison, *J. Chem. Soc.*, 1927, 1723.

Cohen, Kostanecki, *Ber.*, 1904, 37, 2628.

2 : 2' : 4' : 5' - Tetrahydroxychalkone (2:4:5-Trihydroxyphenyl 2-hydroxystyryl ketone).

2' : 4' : 5' - *Tri-Me ether*: green needles from EtOH. M.p. 159-60° decomp. Conc.  $H_2SO_4 \rightarrow$  orange sol. *Acetyl*: golden needles from EtOH.Aq. M.p. 121°.

Reigrodski, Tambor, *Ber.*, 1910, 43, 1967.

2 : 2' : 4' : 6' - Tetrahydroxychalkone (2:4:6-Trihydroxyphenyl 2-hydroxystyryl ketone).

2 : 4' : 6' - *Tri-Me ether*: yellow cryst. from boiling MeOH. M.p. 106-8°. Conc.  $H_2SO_4 \rightarrow$  orange-red col.

Bargellini, Peratoner, *Gazz. chim. ital.*, 1919, 49, ii, 67.

3 : 2' : 3' : 4' - Tetrahydroxychalkone (2:3:4-Trihydroxyphenyl 3-hydroxystyryl ketone).

Yellow needles from 80% MeOH. M.p. 219-20°. Conc.  $H_2SO_4 \rightarrow$  deep orange sol. Alkalis  $\rightarrow$  deep brown sols.

3 : 3' : 4' - *Tri-Me ether*: yellow needles from EtOH. M.p. 127-8°. Conc.  $H_2SO_4 \rightarrow$  orange-yellow sol. *Acetyl*: yellowish needles from EtOH. M.p. 80-1°.

Ellison, *J. Chem. Soc.*, 1927, 1723.

Kostanecki, Schleifenbaum, *Ber.*, 1904, 37, 2631.

4 : 2' : 3' : 4' - Tetrahydroxychalkone (2:3:4-Trihydroxyphenyl 4-hydroxystyryl ketone).

3' : 4' - *Di-Me ether*: yellow needles from EtOH.Aq. M.p. 106°.

4 : 3' : 4' - *Tri-Me ether*: yellow plates. M.p. 131-2°. Conc.  $H_2SO_4 \rightarrow$  orange-yellow sol. *Acetyl*: yellowish prisms from EtOH.Aq. M.p. 89-90°.

*Tetra-Me ether*: pale yellow needles from EtOH. M.p. 94°.

Russell, *J. Chem. Soc.*, 1934, 220.

Kuroda, Matsukuma, *Chem. Zentr.*, 1932, I, 2169.

Kostanecki, Schreiber, *Ber.*, 1905, 38, 2749.

4 : 2' : 4' : 5' - Tetrahydroxychalkone (2:4:5-Trihydroxyphenyl 4-hydroxystyryl ketone).

4 : 4' : 5' - *Tri-Me ether*: red needles from

EtOH.Aq. M.p. 130°. Sol. EtOH,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  orange-yellow sol.

*Tetra-Me ether*: yellow needles from EtOH. M.p. 123-4°. Conc.  $H_2SO_4 \rightarrow$  intense red sol.

Kuroda, Matsukuma, *Chem. Zentr.*, 1932, I, 2169.

Bargellini, Aureli, *Atti accad. Lincei*, 1911, 20, II, 123.

Bargellini, Aurutin, *Gazz. chim. ital.*, 1910, 40, ii, 346.

4 : 2' : 4' : 6' - Tetrahydroxychalkone (2:4:6-Trihydroxyphenyl 4-hydroxystyryl ketone).

4' : 6' - *Di-Me ether*: orange-red needles from  $C_6H_6$ . M.p. 188°. Conc.  $H_2SO_4 \rightarrow$  dark yellow sol. *Diacetyl*: pale yellow needles from MeOH. M.p. 147°.

4 : 4' : 6' - *Tri-Me ether*: yellow needles from EtOH. M.p. 113°. Conc.  $H_2SO_4 \rightarrow$  orange sol. *Acetyl*: yellowish leaflets from EtOH. M.p. 120°.

2' : 4' : 6' - *Tri-Me ether*: golden-yellow cryst. from MeOH. M.p. 195-6°. Conc.  $H_2SO_4 \rightarrow$  orange-red sol. *Acetyl*: yellow cryst. from MeOH. M.p. 108°.

*Tetra-Me ether*: pale yellow cryst. from EtOH.Aq. M.p. 119-21°. Conc.  $H_2SO_4 \rightarrow$  orange-red sol.

4-Carbomethoxyl: yellow cryst. from MeOH.Aq. M.p. 166°.

Mosimann, Tambor, *Ber.*, 1916, 49, 1701.

Kuroda, Matsukuma, *Chem. Zentr.*, 1932, I, 2169.

Bargellini, *Gazz. chim. ital.*, 1914, 44, ii, 424.

Kostanecki, Tambor, *Ber.*, 1904, 37, 792.

2' : 3' : 4' : 6' - Tetrahydroxychalkone (2 : 3 : 4 : 6-Tetrahydroxyphenyl styryl ketone).

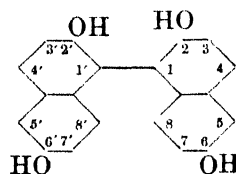
*Tetra-Me ether*: yellowish needles from EtOH.Aq. M.p. 74-5°. Sol.  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  orange-red sol.

Bargellini, Bini, *Gazz. chim. ital.*, 1911, 41, ii, 18.

1 : 3 : 4 : 5 - Tetrahydroxycyclohexane-1-carboxylic Acid.

See Quinic Acid.

2 : 6 : 2' : 6' - Tetrahydroxy-1 : 1' - dinaphthyl



$C_{20}H_{14}O_4$

MW, 318

**3 : 4 : 3' : 4' -Tetrahydroxy-1 : 1'-dinaphthyl**

M.p. 318–20°. Autoxidises readily, especially in alk. sol.

Ioffe, Kusnetzow, *Chem. Zentr.*, 1936, I, 2935.

**3 : 4 : 3' : 4' -Tetrahydroxy-1 : 1'-dinaphthyl.**

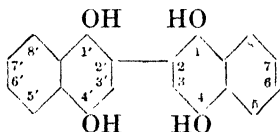
Needles from 30% AcOH. M.p. 205–10° decomp. (176–8°). Sol. AcOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Insol. H<sub>2</sub>O.

*Tetra-Me ether*: C<sub>24</sub>H<sub>22</sub>O<sub>4</sub>. MW, 374. Leaflets from EtOH. M.p. 145–7°. B.p. 200–20°/0.67 mm. Sol. most org. solvents. Spar. sol. EtOH, pet. ether. Conc. H<sub>2</sub>SO<sub>4</sub> → bluish-violet col.

*Tetra-acetyl*: needles from AcOH. M.p. 165–6° decomp. Sol. usual solvents.

Korn, *Ber.*, 1884, 17, 3025.

Strauss, Bernouilly, Mautner, *Ann.*, 1925, 444, 190.

**1 : 4 : 1' : 4' -Tetrahydroxy-2 : 2'-dinaphthyl**

C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>

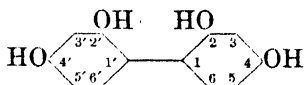
MW, 318

Needles from AcOH. Blackens at 205°. Spar. sol. cold EtOH. Insol. H<sub>2</sub>O. Alc. sol. turns violet in air.

*Tetra-acetyl*: needles from AcOH. M.p. 226–7°.

Meldola, Hughes, *J. Chem. Soc.*, 1890, 57, 632.

See also Liebermann, Schlossberg, *Ber.*, 1899, 32, 546.

**2 : 4 : 2' : 4' -Tetrahydroxydiphenyl**

C<sub>12</sub>H<sub>10</sub>O<sub>4</sub>

MW, 218

Needles from boiling H<sub>2</sub>O. M.p. 226–7°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, AcOEt.

*Tetra-Me ether*: C<sub>16</sub>H<sub>18</sub>O<sub>4</sub>. MW, 274. Cryst. from MeOH. M.p. 93°.

*Tetra-acetyl*: needles from Et<sub>2</sub>O. M.p. 120°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>.

Meyer, Desamari, *Ber.*, 1909, 42, 2823.

Bayer, D.R.P., 90,341.

**2 : 4 : 2' : 5' -Tetrahydroxydiphenyl (Resorcyldydroquinone).**

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 131° under CO<sub>2</sub>.

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**2 : 5 : 2' : 5' -Tetrahydroxydiphenyl sulphide**

Very sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Mod. sol. C<sub>6</sub>H<sub>6</sub>, ligroin. Spar. sol. CS<sub>2</sub>. Alkalis → olive-green sols.

*Tetra-acetyl*: needles from ligroin. M.p. 120° under CO<sub>2</sub>. Sol. CHCl<sub>3</sub>, AcOEt, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, Et<sub>2</sub>O, ligroin, hot H<sub>2</sub>O. Insol. cold NaOH.

Pummerer, Huppmann, *Ber.*, 1927, 60, 1446.

**2 : 4 : 3' : 4' -Tetrahydroxydiphenyl.**

See Sappanin.

**2 : 5 : 2' : 5' -Tetrahydroxydiphenyl (Dihydroquinone).**

Colourless prisms. M.p. 237° decomp. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, hot H<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. FeCl<sub>3</sub> → red col.

*Tetra-Me ether*: C<sub>16</sub>H<sub>18</sub>O<sub>4</sub>. MW, 274. M.p. 104°. Sol. AcOH, C<sub>6</sub>H<sub>6</sub>, hot EtOH. Spar. sol. Et<sub>2</sub>O.

Barth, Schreider, *Monatsh.*, 1884, 5, 590.

Ullmann, Loewenthal, *Ann.*, 1904, 332, 69.

**3 : 4 : 3' : 4' -Tetrahydroxydiphenyl (Dicatechol).**

Cryst. from H<sub>2</sub>O. M.p. 229–30°.

*Tetra-Me ether*: diveratrol. Needles from MeOH.Aq. M.p. 133°. Conc. H<sub>2</sub>SO<sub>4</sub> → golden-yellow sol. → olive-green col. on warming.

Seer, Karl, *Monatsh.*, 1913, 34, 647.

Späth, Gibian, *Monatsh.*, 1930, 55, 347.

**3 : 5 : 3' : 5' -Tetrahydroxydiphenyl (Diresorcinol).**

Needles + 2H<sub>2</sub>O from hot H<sub>2</sub>O. M.p. anhyd. 310°. Mod. sol. hot H<sub>2</sub>O. Prac. insol. AcOH. Zn dust dist. → diphenyl. FeCl<sub>3</sub> → light blue col.

*Tetra-Me ether*: white cryst. M.p. 108°.

*Tetra-Et ether*: C<sub>20</sub>H<sub>26</sub>O<sub>4</sub>. MW, 330. Leaflets. M.p. 110°. Sol. Et<sub>2</sub>O.

*Tetra-acetyl*: m.p. 157–9°.

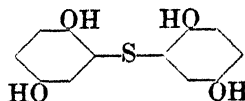
Benedikt, Julius, *Monatsh.*, 1884, 5, 177.

v. Friedrichs, *Chem. Zentr.*, 1916, I, 975.

**2 : 6 : 2' : 6' -Tetrahydroxydiphenyl.**

*Tetra-Me ether*: cryst. from Me<sub>2</sub>CO.Aq. M.p. 175–6°.

v. Arendonk, Cupery, Adams, *J. Am. Chem. Soc.*, 1933, 55, 4227.

**2 : 5 : 2' : 5' -Tetrahydroxydiphenyl sulphide**

C<sub>12</sub>H<sub>10</sub>O<sub>4</sub>S

MW, 250

Cryst. M.p. 227-9°.

*Tetra-Me ether*:  $C_{16}H_{18}O_4S$ . MW, 306.  
Amorph. M.p. 97-100°. Sol.  $Me_2CO$ , hot EtOH.

Badische, D.R.P., 175,070, (*Chem. Zentr.*, 1906, I, 1466).

Smiles, Le Rossignol, *J. Chem. Soc.*, 1908, 93, 760.

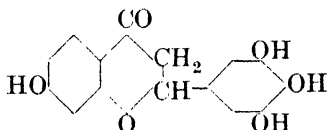
**5 : 6 : 7 : 4'-Tetrahydroxyflavanone.**

See Isocarthamidin.

**5 : 7 : 8 : 4'-Tetrahydroxyflavanone.**

See Carthamidin.

**7 : 3' : 4' : 5'-Tetrahydroxyflavanone**



$C_{15}H_{12}O_6$

MW, 288

*Tetra-Me ether*:  $C_{19}H_{20}O_6$ . MW, 344.  
Needles from EtOH. M.p. 148-9°. Conc.  $H_2SO_4$  → yellow sol.

Dean, Nierenstein, *J. Am. Chem. Soc.*, 1925, 47, 1681.

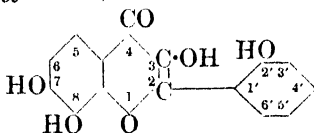
**3 : 5 : 7 : 2'-Tetrahydroxyflavone.**

See Datisectin.

**3 : 5 : 7 : 4'-Tetrahydroxyflavone.**

See Kämpferol.

**3 : 7 : 8 : 2'-Tetrahydroxyflavone (7 : 8 : 2'-Trihydroxyflavonol)**



$C_{15}H_{10}O_6$

MW, 286

Pale yellow needles. M.p. 298° decomp. NaOH → reddish-yellow sol.

*7 : 8 : 2'-Tri-Me ether*:  $C_{18}H_{16}O_6$ . MW, 328.  
Cryst. from EtOH. M.p. 212-14°. NaOH → yellow sol. Conc.  $H_2SO_4$  → greenish-yellow sol. *Acetyl*: needles from EtOH.Aq. M.p. 138-9°.

Cohen, Kostanecki, *Ber.*, 1904, 37, 2630.

**3 : 7 : 8 : 3'-Tetrahydroxyflavone (7 : 8 : 3'-Trihydroxyflavonol).**

Pale yellow needles from EtOH.Aq. M.p. 260°. Conc.  $H_2SO_4$  → greenish-yellow sol. Alkalis → brownish-yellow sols.

*7 : 8 : 3'-Tri-Me ether*: pale yellow needles. M.p. 188-9°. Spar. sol. EtOH. Conc.  $H_2SO_4$  → pale yellow sol. *Acetyl*: needles from EtOH.Aq. M.p. 165°.

*Tetra-acetyl*: needles from EtOH.Aq. M.p. 166-7°.

Kostanecki, Schleifenbaum, *Ber.*, 1904, 37, 2633.

**3 : 7 : 8 : 4'-Tetrahydroxyflavone (7 : 8 : 4'-Trihydroxyflavonol).**

Pale yellow needles +  $H_2O$  from EtOH. M.p. anhyd. 319° decomp. Dil. NaOH → orange-red sol. Conc.  $H_2SO_4$  → yellow sol. with weak green fluor.

*7 : 8 : 4'-Tri-Me ether*: pale yellow needles from EtOH. M.p. 198°. Conc.  $H_2SO_4$  → pale yellow sol. *Acetyl*: needles from EtOH.Aq. M.p. 157°.

*Tetra-acetyl*: needles from EtOH.Aq. M.p. 175°.

Kostanecki, Schreiber, *Ber.*, 1905, 38, 2751.

**5 : 6 : 7 : 4'-Tetrahydroxyflavone.**

See Scutellarein.

**5 : 7 : 8 : 4'-Tetrahydroxyflavone.**

Yellow needles from AcOH.Aq. M.p. 247-8°. Alkalis → reddish-brown sols.  $FeCl_3$  → greenish-brown col.

*5 : 8 : 4'-Tri-Me ether*: yellow cryst. from EtOH. M.p. 279-80° (258°). *Acetyl*: m.p. 194-5°.

*Tetra-Me ether*: needles. M.p. 207-8°.

*Tetra-acetyl*: needles from EtOH. M.p. 252-3°.

Furukawa, Tamaki, *Chem. Abstracts*, 1932, 26, 142.

Hattori, *Acta Phytochimica*, 1931, 5, 219.

Wessely, *Monatsh.*, 1930, 56, 97.

**3 : 6 : 2' : 4'-Tetrahydroxyflavone (6 : 2' : 4'-Trihydroxyflavonol).**

Pale yellow needles +  $H_2O$  from EtOH.Aq. M.p. anhyd. 285°. Dil. NaOH → greenish-yellow sol. with greenish fluor. Conc.  $H_2SO_4$  → pale yellow sol. with weak green fluor.

*6 : 2' : 4'-Tri-Me ether*: pale yellow cryst. from EtOH or  $C_6H_6$ . M.p. 193°. Conc.  $H_2SO_4$  → greenish-yellow sol. with light green fluor. *Acetyl*: prismatic needles from EtOH.Aq. M.p. 162°.

*Tetra-acetyl*: needles from EtOH.Aq. M.p. 163°.

Bonifazi, Kostanecki, Tambor, *Ber.*, 1906, 39, 90.

**3 : 6 : 3' : 4'-Tetrahydroxyflavone (6 : 3' : 4'-Trihydroxyflavonol).**

Yellow needles from EtOH. M.p. 335° decomp. Alc. sol. shows greenish fluor. NaOH

**3 : 7 : 2' : 4'-Tetrahydroxyflavone**

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→ reddish-yellow sol. Conc.  $\text{H}_2\text{SO}_4$  → orange sol. with greenish fluor.

6 : 3' : 4'-*Tri-Me ether* : yellowish needles from EtOH. M.p. 189–90°. Conc.  $\text{H}_2\text{SO}_4$  → yellow sol. with greenish fluor. *Acetyl* : needles from EtOH.Aq. M.p. 140–1°.

*Tetra-acetyl* : needles from EtOH or AcOH-EtOH. M.p. 197–8°. Spar. sol. EtOH.

Kostanecki, Kugler, *Ber.*, 1904, **37**, 781.  
Auwers, Pohl, *Ann.*, 1914, **405**, 287.

**3 : 7 : 2' : 4'-Tetrahydroxyflavone.**

See Resomorin.

**3 : 7 : 3' : 4'-Tetrahydroxyflavone.**

See Fisetin.

**5 : 7 : 2' : 4'-Tetrahydroxyflavone.**

Needles from EtOH or AcOH. M.p. 332–5° (277°) decomp. Conc.  $\text{H}_2\text{SO}_4$  → pale yellow sol. with violet-blue fluor.

2' : 4'-*Di-Me ether* : pale yellow needles from AcOH.Aq. + HCl. M.p. 258–9°. Mod. sol. boiling EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ . Alc.  $\text{FeCl}_3$  → weak greenish-brown col. Alkalis → bright yellow sols. Conc.  $\text{H}_2\text{SO}_4$  → pale yellow sol. with blue fluor.

Robinson, Venkataraman, *J. Chem. Soc.*, 1929, 65.

**5 : 7 : 3' : 4'-Tetrahydroxyflavone.**

See Luteolin.

**7 : 8 : 3' : 4'-Tetrahydroxyflavone.**

Yellow needles from EtOH.Aq. M.p. 309–10° decomp. Conc.  $\text{H}_2\text{SO}_4$  → pale yellow col. with no fluor. NaOH → orange-red sol. Alc.  $\text{FeCl}_3$  → dark green col.

*Tetra-Me ether* : yellow needles from EtOH. M.p. 198–9°.

*Tetra-acetyl* : needles from EtOH. M.p. 218°.

Badhwar, Kang, Venkataraman, *J. Chem. Soc.*, 1932, 1109.

**6 : 3' : 4' : 5'-Tetrahydroxyflavone.**

Yellow needles from EtOH.Aq. M.p. 347°. Conc.  $\text{H}_2\text{SO}_4$  → bright yellow sol. with no fluor. NaOH → bright red sol. Alc.  $\text{FeCl}_3$  → intense green col.

3' : 4' : 5'-*Tri-Me ether* : pale orange plates from EtOH.Aq. M.p. 232–3°. No col. with  $\text{FeCl}_3$ . Conc.  $\text{H}_2\text{SO}_4$  → bright yellow sol. with weak green fluor. *Acetyl* : needles from EtOH. M.p. 185°.

*Tetra-acetyl* : needles from EtOH-AcOH. M.p. 258–9°.

Chadha, Venkataraman, *J. Chem. Soc.*, 1933, 1075.

**1 : 3 : 5 : 8-Tetrahydroxy-2-methyl-anthraquinone****7 : 2' : 4' : 6'-Tetrahydroxyflavone.**

Yellow plates from EtOH. M.p. 240°. Spar. sol.  $\text{CHCl}_3$ . Conc.  $\text{H}_2\text{SO}_4$  → yellow sol. with greenish fluor.

Cullinane, Algar, Ryan, *Chem. Abstracts*, 1929, **23**, 4472.

**7 : 3' : 4' : 5'-Tetrahydroxyflavone.**

Needles +  $\text{H}_2\text{O}$  from EtOH.Aq. M.p. 340° decomp. Mod. sol. hot EtOH. NaOH → orange-red sol. Conc.  $\text{H}_2\text{SO}_4$  → yellow sol. with weak greenish-yellow fluor.

*Tetra-Me ether* : needles. M.p. 191–2°. Mod. sol. boiling EtOH with violet fluor. Conc.  $\text{H}_2\text{SO}_4$  → yellowish sol. with weak green fluor.

*Tetra-acetyl* : leaflets from EtOH. M.p. 215°.

Kostanecki, Plattner, *Ber.*, 1902, **35**, 2546.

**1 : 3 : 4 : 5 - Tetrahydroxyhexahydro-benzoic Acid.**

See Quinic Acid.

**Tetrahydroxyisovaleraldehyde.**

See Apiose.

**Tetrahydroxyisovaleric Acid.**

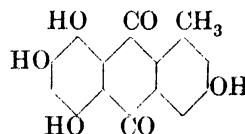
See Apionic Acid.

**Tetrahydro-xylene.**

See Dimethylcyclohexene.

**Tetrahydroxymethane.**

See Orthocarbonic Acid.

**3 : 5 : 7 : 8 - Tetrahydroxy - 1 - methyl-anthraquinone**

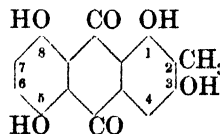
$\text{C}_{15}\text{H}_{10}\text{O}_6$

MW, 286

Light red needles from EtOH or Py. Sol. EtOH, AcOH, Py. Spar. sol.  $\text{C}_6\text{H}_6$ .

*Tetra-acetyl* : m.p. 185–6°.

Dimroth, Fick, *Ann.*, 1916, **411**, 325.

**1 : 3 : 5 : 8 - Tetrahydroxy - 2 - methyl-anthraquinone**

$\text{C}_{15}\text{H}_{10}\text{O}_6$

MW, 286

Yellowish-red plates from 50% AcOH. M.p. 276–7°.

**1 : 4 : 5 : 8-Tetrahydroxy-2-methyl-anthraquinone**

*Tetra-acetyl*: yellow plates from AcOH. M.p. 223°.

Charlesworth, Robinson, *J. Chem. Soc.*, 1934, 1531.

**1 : 4 : 5 : 8 - Tetrahydroxy - 2 - methyl - anthraquinone (*Cynodontin*).**

Found in *Helminthosporium cynodontis*, M., and *Helminthosporium eualacnæ*, Z. Cryst. from Py. M.p. 260°. Sol. most org. solvents. NaOH  $\longrightarrow$  deep bluish-violet sol. Conc. H<sub>2</sub>SO<sub>4</sub>  $\longrightarrow$  blue sol. with red fluor.

*Tetra-acetyl*: m.p. 224–5°.

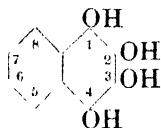
Raistrick, Robinson, Todd, *Biochem. J.*, 1933, 27, 1170.

I.C.I., E.P., 420,362; F.P., 770,972, (*Chem. Abstracts*, 1935, 29, 816).

**Tetrahydroxymethyl-methane.**

See Pentaerythritol.

**1 : 2 : 3 : 4 - Tetrahydroxynaphthalene (*Leucoisonaphthazarin*)**



C<sub>10</sub>H<sub>8</sub>O<sub>4</sub>

MW, 192

Cryst. from C<sub>6</sub>H<sub>6</sub>, leaflets from Et<sub>2</sub>O-pet. ether. M.p. 225°. Sol. H<sub>2</sub>O, most org. solvents. Spar. sol. C<sub>6</sub>H<sub>6</sub>, pet. ether. Easily decomp.

*Tetra-acetyl*: needles from AcOH. M.p. 220°. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>.

Zincke, Ossenbeck, *Ann.*, 1899, 307, 16.

Leeds, *J. Am. Chem. Soc.*, 1880, 2, 285.

**1 : 2 : 4 : 6-Tetrahydroxynaphthalene.**

*Tetra-acetyl*: leaflets from AcOH. M.p. 181–2°.

Dimroth, Kerkovius, *Ann.*, 1913, 399, 39.

**1 : 2 : 4 : 7-Tetrahydroxynaphthalene.**

*Tetra-acetyl*: cryst. from ligroin. M.p. 140–1°.

See previous reference.

**1 : 4 : 5 : 8 - Tetrahydroxynaphthalene (*Leuconaphthazarin*).**

Yellow plates or prisms from HCl. M.p. 153–4°. Mod. sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, pet. ether. Turns red in air.

*Tetra-Me ether*: C<sub>14</sub>H<sub>16</sub>O<sub>4</sub>. MW, 248. Cryst. from AcOH. M.p. about 170°. Sol. AcOH, toluene, xylene.

1:4-*Diacetyl*: plates from AcOEt. M.p. 241–3°. Very sol. Me<sub>2</sub>CO. Sol. EtOH. Insol. CHCl<sub>3</sub>, xylene.

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**2 : 3 : 4 : 5-Tetrahydroxytoluene**

*Tetra-acetyl*: leaflets from AcOH. M.p. 277–9° decomp. Sol. hot AcOH. Spar. sol. other solvents.

Zincke, Schmidt, *Ann.*, 1895, 286, 37.

Badische, D.R.P., 129,074, (*Chem. Zentr.*, 1902, I, 691).

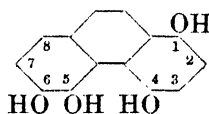
Perkin, Weizmann, *J. Chem. Soc.*, 1906, 89, 1658.

Wheeler, Edwards, *J. Am. Chem. Soc.*, 1916, 38, 387; 1917, 39, 2465.

**2 : 3 : 4 : 6-Tetrahydroxyphenacyl Alcohol.**

See Gossypitol.

**1 : 4 : 5 : 6-Tetrahydroxyphenanthrene**



C<sub>14</sub>H<sub>10</sub>O<sub>4</sub>

MW, 242

*Tetra-Me ether*: C<sub>18</sub>H<sub>18</sub>O<sub>4</sub>. MW, 298. Light brown needles from MeOH. M.p. 118–20°. *Picrate*: dark chocolate needles from EtOH. M.p. 158°.

Gulland, Virden, *J. Chem. Soc.*, 1928, 1486.

**2 : 3 : 5 : 6-Tetrahydroxyphenanthrene.**

2:6-*Di-Me ether*: sinomenol. C<sub>16</sub>H<sub>14</sub>O<sub>4</sub>. MW, 270. M.p. 176°. *Diacetyl*: m.p. 206°.

*Tetra-Me ether*: cryst. from EtOH. M.p. 124–5°. *Picrate*: m.p. 123–5°.

Kondo, Ochiai, *Chem. Abstracts*, 1928, 22, 4532.

Goto, *Chem. Abstracts*, 1927, 21, 1654.

**Tetrahydroxyphenyl styryl Ketone.**

See 2' : 3' : 4' : 6'-Tetrahydroxychalkone.

**8 : 9 : 11 : 12-Tetrahydroxystearic Acid.**

See Sativic Acid.

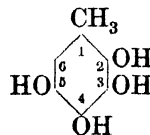
**Tetrahydroxysuccinic Acid.**

See Diketosuccinic Acid.

**Tetrahydroxytetramethylmethane.**

See Pentaerythritol.

**2 : 3 : 4 : 5 - Tetrahydroxytoluene (5-Methylapionol)**



C<sub>7</sub>H<sub>8</sub>O<sub>4</sub>

MW, 156

Cryst. from toluene. M.p. 170–1°. Sol. H<sub>2</sub>O, EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>, toluene. NaOH  $\longrightarrow$  intense green col.  $\longrightarrow$  blue on shaking in air.



4-Me ether :  $C_8H_{10}O_4$ . MW, 170. Triacetyl : m.p. 91-2°.

Tetra-Me ether :  $C_{11}H_{16}O_4$ . MW, 212. M.p. 51-2°.

Tetra-acetyl : cryst. from MeOH. M.p. 132-3°. Sol. most org. solvents.

Thiele, Winter, *Ann.*, 1900, **311**, 352.

Erdtman, *Chem. Abstracts*, 1934, **28**, 1337.

### 2 : 3 : 4 : 6-Tetrahydroxytoluene.

4-Me ether : cryst. Oxidises at 134°. Sol.  $H_2O$ , AcOEt. Mod. sol. EtOH,  $Et_2O$ . Insol.  $C_6H_6$ , ligroin. Triacetyl : cryst. from AcOEt. M.p. 174°.

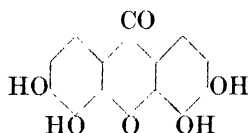
Konya, *Monatsh.*, 1900, **21**, 430.

### 2 : 3 : 5 : 6-Tetrahydroxytoluene.

Tetra-acetyl : needles from EtOH. M.p. 198°.

Fichter, *Ann.*, 1908, **361**, 401.

### 1 : 2 : 8 : 9-Tetrahydroxyxanthone



$C_{13}H_8O_6$

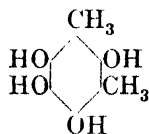
MW, 260

Light brown cryst. powder. Sol. EtOH,  $Me_2CO$ . Spar. sol. hot  $H_2O$ . Insol.  $CHCl_3$ ,  $C_6H_6$ .  $NaOH \rightarrow$  yellowish-brown sol.

Tetra-acetyl : cryst. from  $C_6H_6$ . M.p. 237°.

Buchka, *Ann.*, 1881, **209**, 270.

### 2 : 4 : 5 : 6-Tetrahydroxy-m-xylene



$C_8H_{10}O_4$

MW, 170

Needles from  $Et_2O$ -ligroin. M.p. 189° (rapid heat.). Sol.  $H_2O$ , EtOH,  $Et_2O$ . Insol. ligroin. Conc.  $H_2SO_4 \rightarrow$  green col.  $\rightarrow$  bluish-violet on dilution.

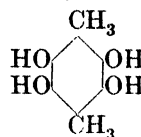
4-Me ether : prisms from  $C_6H_6$ . M.p. 125°. Triacetyl : cryst. from EtOH. M.p. 76°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin.

Tetra-acetyl : needles from boiling EtOH. M.p. 154°. Sol.  $Et_2O$ . Insol. cold  $H_2O$ , EtOH.

Brunnmayr, *Monatsh.*, 1900, **21**, 10.

Bosse, *ibid.*, 1028.

### 2 : 3 : 5 : 6-Tetrahydroxy-p-xylene



$C_8H_{10}O_4$

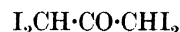
MW, 170

2 : 5-Diacetyl : needles from AcOH. M.p. 223°.

Tetra-acetyl : needles from EtOH. M.p. 242°.

Fichter, *Ann.*, 1908, **361**, 378.

### sym.-Tetraiodoacetone



$C_2H_2OI_4$

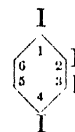
MW, 562

Yellow needles. M.p. 142°. Very sol.  $Me_2CO$ .

Angeli, Levi, *Gazz. chim. ital.*, 1893, **23**, ii, 97.

Lederer, D.R.P., 95,440.

### 1 : 2 : 3 : 4-Tetraiodobenzene



$C_6H_2I_4$

MW, 582

Prisms from  $CS_2$  or  $Et_2O$ -AcOH. M.p. 136° (114°). Sublimes. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

Willgerodt, Arnold, *Ber.*, 1901, **34**, 3353.

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 688.

### 1 : 2 : 3 : 5-Tetraiodobenzene.

Cryst. from AcOH or  $Et_2O$ . M.p. 148°. Sublimes. Sol. boiling AcOH. Spar. sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

Willgerodt, Arnold, *Ber.*, 1901, **34**, 3350.

### 1 : 2 : 4 : 5-Tetraiodobenzene.

Needles from  $Et_2O$ , prisms from  $C_6H_6$ . M.p. 254° (165°). Sol. AcOH,  $CS_2$ . Very spar. sol. EtOH,  $Et_2O$ . Sublimes in vacuo.

Willgerodt, Arnold, *Ber.*, 1901, **34**, 3352.

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 687.

### Tetraiodobenzoquinone.

See Iodanil.

### Tetraiodoethylene (Periodoethylene)



$C_2I_4$

MW, 532

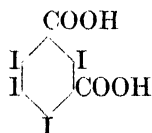
Lemon-yellow leaflets from AcOH. M.p. 192° (187°).  $D^{20}$  2.983. Very sol.  $CS_2$ .

Spar. sol. abs. EtOH. Forms add. comps. with amines. Dissociates in sunlight  $\rightarrow \text{I}_2 + \text{C}_2\text{I}_2$ .

Datta, Prosad, *J. Am. Chem. Soc.*, 1917, 39, 451.

Biltz, Küppers, *Ber.*, 1904, 37, 4415.

#### Tetraiodoisophthalic Acid



$\text{C}_8\text{H}_2\text{O}_4\text{I}_4$  MW, 670

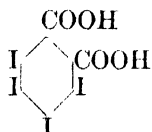
Prisms from AcOH. M.p. 308–12° decomp. Sol. MeOH. Very spar. sol.  $\text{Et}_2\text{O}$ , AcOH.

Rupp, *Ber.*, 1896, 29, 1632.

#### Tetraiodomethane.

See Carbon tetraiodide.

#### Tetraiodophthalic Acid



$\text{C}_8\text{H}_2\text{O}_4\text{I}_4$  MW, 670

Needles from  $\text{PhNO}_2$ . M.p. 324–7°. Very spar. sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH.

*Mono-Me ester*:  $\text{C}_9\text{H}_4\text{O}_4\text{I}_4$ . MW, 684. Yellow cryst. from AcOH. M.p. 298° decomp.

*Anhydride*:  $\text{C}_8\text{O}_3\text{I}_4$ . MW, 652. Yellow needles from  $\text{PhNO}_2$ . M.p. 325° (320–5°).

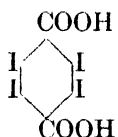
Rupp, *Ber.*, 1896, 29, 1634.

Pratt, Shup, *J. Am. Chem. Soc.*, 1918, 40, 254.

#### Tetraiodopyrrole.

See Iodol.

#### Tetraiodoterephthalic Acid



$\text{C}_8\text{H}_2\text{O}_4\text{I}_4$  MW, 670

Prisms from AcOH. M.p. 315–20° decomp. Sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ , AcOH,  $\text{C}_6\text{H}_6$ .

*Di-Me ester*:  $\text{C}_{10}\text{H}_6\text{O}_4\text{I}_4$ . MW, 698. Cryst. from xylene. M.p. 310–12°. Very spar. sol.  $\text{H}_2\text{O}$ .

*Di-Et ester*:  $\text{C}_{12}\text{H}_{10}\text{O}_4\text{I}_4$ . MW, 726. Cryst. from xylene. M.p. 262–5°.

*Dipropyl ester*:  $\text{C}_{14}\text{H}_{14}\text{O}_4\text{I}_4$ . MW, 754. M.p. 239°.

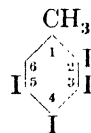
Dict. of Org. Comp.—III.

*Dichloride*:  $\text{C}_8\text{O}_2\text{Cl}_2\text{I}_4$ . MW, 707. Cryst. from  $\text{CHCl}_3$ . M.p. 279°. Very stable towards NaOH.

Lütjens, *Ber.*, 1896, 29, 2836.

Rupp, *ibid.*, 1629.

#### 2 : 3 : 4 : 5-Tetraiodotoluene



$\text{C}_7\text{H}_4\text{I}_4$  MW, 596

Yellow needles from  $\text{C}_6\text{H}_6$ . M.p. 284–5°. Mod. sol.  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, 44, 506.

#### 2 : 3 : 4 : 6-Tetraiodotoluene.

Needles from  $\text{C}_6\text{H}_6$ . M.p. 170°. Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, 44, 133.

#### 2 : 3 : 5 : 6-Tetraiodotoluene.

Needles from  $\text{C}_6\text{H}_6$ . M.p. 125°. Sol.  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, 44, 499.

#### 1 : 3 : 4 : 6-Tetraketo-1 : 6-diphenylhexane.

See Diphenacyl Diketone.

#### Tetralin.

See Tetrahydronaphthalene.

#### Tetralol.

See Tetrahydronaphthol.

#### Tetralone.

See Ketotetrahydronaphthalene.

#### 2 : 3 : 4 : 6-Tetramethylacetanilide.

See under Isoduridine.

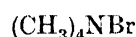
#### Tetramethylacetone.

Di-isopropyl Ketone, *q.v.*

#### Tetramethylalloxantin.

See Amalic Acid.

#### Tetramethylammonium bromide



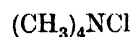
$\text{C}_4\text{H}_{12}\text{NBr}$  MW, 154

Volatile cryst. Sublimes with decomp. above 230°. Sol.  $\text{H}_2\text{O}$ . Spar. sol. abs. EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

Duvillier, Buisine, *Ann. chim. phys.*, 1881, 23, 327.

Schmidt, *Ann.*, 1892, 267, 265.

#### Tetramethylammonium chloride

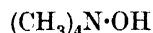


$\text{C}_4\text{H}_{12}\text{NCl}$  MW, 109.5

Volatile cryst. Decomp. above 230° to trimethylamine + CH<sub>3</sub>Cl. Sol. H<sub>2</sub>O, EtOH. Insol. CHCl<sub>3</sub>.

Vincent, Chappuis, *Bull. soc. chim.*, 1886, 45, 502.

## Tetramethylammonium hydroxide

C<sub>4</sub>H<sub>13</sub>ON

MW, 91

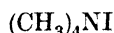
*Pentahydrate* (+ 5H<sub>2</sub>O): hygroscopic needles. M.p. 63°. Decomp. on dist. to trimethylamine and CH<sub>3</sub>OH. Very sol. H<sub>2</sub>O. Absorbs CO<sub>2</sub> rapidly. Gentle warming at 40–50° → *trihydrate* (+ 3H<sub>2</sub>O), m.p. 60°. Careful warming *in vacuo* → *monohydrate* (+ 1H<sub>2</sub>O), decomp. at 130° to trimethylamine and CH<sub>3</sub>OH. Very powerful alkali, comparable with NaOH. The salts form a large number of add. comps. with metal and other halides.

Walker, Johnston, *J. Chem. Soc.*, 1905, 87, 958.

Schmidt, *Ann.*, 1892, 267, 267.

Hofmann, *Ann.*, 1851, 79, 18.

## Tetramethylammonium iodide

C<sub>4</sub>H<sub>12</sub>NI

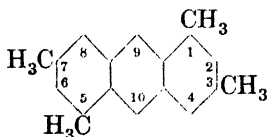
MW, 201

Prisms. Decomp. above 230°. Spar. sol. cold H<sub>2</sub>O, abs. EtOH. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Triboluminescent.

Chablay, *Ann. chim.*, 1914, 1, 477.

Hofmann, *Ann.*, 1851, 79, 16.

## 1 : 3 : 5 : 7-Tetramethylantracene

C<sub>18</sub>H<sub>18</sub>

MW, 234

Yellowish leaflets from EtOH. M.p. 163–4°. *Picrate*: m.p. 189–90°.

Seer, *Monatsh.*, 1912, 33, 42.

## 1 : 3 : 6 : 8-Tetramethylantracene.

Pale yellow plates with green fluor. from AcOH. M.p. 281–3° (280°). Sol. hot EtOH, hot Et<sub>2</sub>O. Mod. sol. CHCl<sub>3</sub>. CrO<sub>3</sub> in AcOH → 1 : 3 : 6 : 8-tetramethylantraquinone.

Seer, *Monatsh.*, 1912, 33, 36.

Anschütz, *Ann.*, 1886, 235, 174.

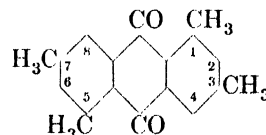
Dewar, Jones, *J. Chem. Soc.*, 1904, 85, 217.

## 2 : 3 : 6 : 7-Tetramethylantracene.

Pale yellow plates from CS<sub>2</sub>. M.p. 301°. Sublimes. Sol. AcOH, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH.

Morgan, Coulson, *J. Chem. Soc.*, 1931, 2331.

## 1 : 3 : 5 : 7-Tetramethylantraquinone

C<sub>18</sub>H<sub>16</sub>O<sub>2</sub>

MW, 264

Yellow needles from EtOH. M.p. 235°. Sol. AcOH, C<sub>6</sub>H<sub>6</sub>. Very spar. sol. EtOH. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with deep red col. Sublimes. HNO<sub>3</sub> (D 1.1) at 190–210° → anthraquinone-1 : 3 : 5 : 7-tetracarboxylic acid. Dist. with Zn dust → 1 : 3 : 5 : 7-tetramethylantracene.

Seer, *Monatsh.*, 1912, 33, 39.

## 1 : 3 : 6 : 8-Tetramethylantraquinone.

M.p. 228–30° (172–3°).

Seer, *Monatsh.*, 1912, 33, 36.

Dewar, Jones, *J. Chem. Soc.*, 1904, 85, 218.

Börnstein, Schlieviensky, Szczesny-Heyl, *Ber.*, 1926, 59, 2815.

## 2 : 3 : 5 : 8-Tetramethylantraquinone.

Yellow needles. M.p. 178°.

Fieser, Fieser, *J. Am. Chem. Soc.*, 1935, 57, 1679.

## 2 : 3 : 6 : 7-Tetramethylantraquinone.

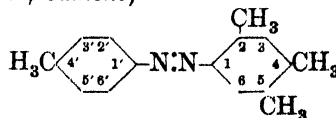
Yellow needles or plates from AcOH. M.p. 330°. Sublimes. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with deep red col.

Morgan, Coulson, *J. Chem. Soc.*, 1931, 2329.

## 2 : 3 : 6 : 8-Tetramethylantraquinone.

Yellow needles. M.p. 196°.

Fieser, Fieser, *J. Am. Chem. Soc.*, 1935, 57, 1679.

2 : 4 : 5 : 4'-Tetramethylazobenzene (5-p-Tolueneazo-*ψ*-cumene)C<sub>16</sub>H<sub>18</sub>N<sub>2</sub>

MW, 238

Yellow needles. M.p. 58°.

Michaelis, Petow, *Ber.*, 1898, 31, 994.

**2:3:2':3'-Tetramethylazobenzene** (3:3'-Azo-o-xylene).

Orange needles. M.p. 110–11°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

Noelting, Stricker, *Ber.*, 1888, **21**, 3139.

**2:4:2':4'-Tetramethylazobenzene** (4:4'-Azo-m-xylene).

Red needles from EtOH. M.p. 129° (126°). Sol. EtOH, Et<sub>2</sub>O.

Noelting, Stricker, *Ber.*, 1888, **21**, 3141.

Vorländer, Meyer, *Ann.*, 1902, **320**, 128.

**2:4:3':5'-Tetramethylazobenzene** (4:5'-Azo-m-xylene).

Red plates or needles from EtOH. M.p. 46–7°. Sol. common org. solvents.

Zincke, Jaenke, *Ber.*, 1888, **21**, 543.

**2:5:2':5'-Tetramethylazobenzene** (2:2'-Azo-p-xylene).

Yellow needles. M.p. 119°.

Noelting, Stricker, *Ber.*, 1888, **21**, 3143.

**3:4:3':4'-Tetramethylazobenzene** (4:4'-Azo-o-xylene).

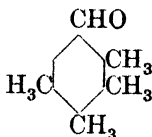
Red needles. M.p. 140–1°.

Noelting, Stricker, *Ber.*, 1888, **21**, 3140.

**3:5:3':5'-Tetramethylazobenzene** (5:5'-Azo-m-xylene).

Orange needles. M.p. 136–7°. Sol. EtOH, Et<sub>2</sub>O.

Noelting, Stricker, *Ber.*, 1888, **21**, 3142.

**2 : 3 : 4 : 5-Tetramethylbenzaldehyde**

C<sub>11</sub>H<sub>14</sub>O

MW, 162

Cryst. M.p. 39°. Very sol. most org. solvents. Oxidises in air. KMnO<sub>4</sub> → 2 : 3 : 4 : 5-tetramethylbenzoic acid.

Semicarbazone: needles from EtOH. M.p. 229–30°.

Auwers, Köckritz, *Ann.*, 1907, **352**, 316.

**1 : 2 : 3 : 4-Tetramethylbenzene.**

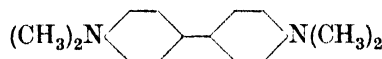
See Prehnitene.

**1 : 2 : 3 : 5-Tetramethylbenzene.**

See Isodurene.

**1 : 2 : 4 : 5-Tetramethylbenzene.**

See Durene.

**N : N'-Tetramethylbenzidine** (4 : 4'-Tetramethyldiaminodiphenyl)

C<sub>16</sub>H<sub>20</sub>N<sub>2</sub>

MW, 240

Needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 198° (193°). B.p. above 360°. Very sol. CHCl<sub>3</sub>. Sol. hot AcOEt, hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O, boiling ligroin. Very spar. sol. MeOH, EtOH. Non-volatile in steam.

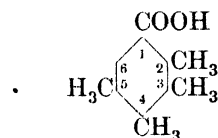
Monomethochloride: m.p. 228°. Sol. H<sub>2</sub>O, EtOH.

Monomethiodide: needles from H<sub>2</sub>O. M.p. 263°.

König, Seifert, *Ber.*, 1934, **67**, 2119.

Willstätter, Kalb, *Ber.*, 1904, **37**, 3765.

Ullmann, Dieterle, *ibid.*, 29.

**2 : 3 : 4 : 5-Tetramethylbenzoic Acid** (Prehnitine-carboxylic acid)

C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>

MW, 178

Needles from EtOH or ligroin. M.p. 168–9° (165°). Volatile in steam.

Me ester: C<sub>12</sub>H<sub>16</sub>O<sub>2</sub>. MW, 192. Needles from CHCl<sub>3</sub>. M.p. 36°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, ligroin.

Amide: C<sub>11</sub>H<sub>15</sub>ON. MW, 177. Needles from EtOH. M.p. 222°. Spar. sol. Et<sub>2</sub>O.

v. Meyer, Molz, *Ber.*, 1897, **30**, 1279.

Smith, Harris, *J. Am. Chem. Soc.*, 1935, **57**, 1292.

**2 : 3 : 4 : 6-Tetramethylbenzoic Acid** (Isodurene-carboxylic acid).

Prisms from ligroin. M.p. 164–5°.

Amide: needles from H<sub>2</sub>O. M.p. 141–2°.

Nitrile: C<sub>11</sub>H<sub>13</sub>N. MW, 159. Cryst. from EtOH. M.p. 68–9°. Stable to HCl at 200°. HCl at 250° → isodurene.

Gattermann, *Ber.*, 1899, **32**, 1118.

**2 : 3 : 5 : 6-Tetramethylbenzoic Acid** (Durene-carboxylic acid).

Prisms from H<sub>2</sub>O or ligroin. M.p. 179° (176.5°). Sol. EtOH, boiling H<sub>2</sub>O. Volatile in steam. Sublimes. HCl at 200–20° → durene.

Me ester: leaflets from EtOH. M.p. 59°.

Amide: cryst. from H<sub>2</sub>O. M.p. 178°.

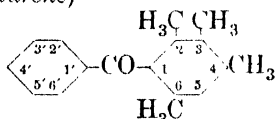
*Nitrile*: needles from EtOH. M.p. 76-7°. Conc. HCl at 210-20° → durene.

Jacobsen, *Ber.*, 1889, **22**, 1223.

Gattermann, *Ber.*, 1899, **32**, 1119.

Meyer, Wöhler, *Ber.*, 1896, **29**, 2572.

**2 : 3 : 4 : 6 - Tetramethylbenzophenone**  
(*Benzoylisodurene*)



$C_{17}H_{18}O$

MW, 238

Cryst. M.p. 62-3°. B.p. about 300°.

Essner, Gossin, *Bull. soc. chim.*, 1884, **42**, 171.

**2 : 3 : 5 : 6 - Tetramethylbenzophenone**  
(*Benzoyldurene*).

M.p. 119°. B.p. 343-343.5°/725 mm. Very sol. hot EtOH. KOH fusion → durene + benzoic acid.

Friedel, Crafts, Ador, *Ann. chim. phys.*, 1884, **1**, 511.

**2 : 4 : 5 : 4' - Tetramethylbenzophenone.**

Viscous oil. B.p. 220°/22 mm.

*Oxime*: cryst. from EtOH. M.p. 151°.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2554.

**2 : 4 : 2' : 4' - Tetramethylbenzophenone.**

B.p. 190°/10 mm., 188°/7 mm.  $D_4^{20}$  1.043.  $n_D^{15}$  1.5876. Does not react with hydroxylamine or phenylhydrazine. Zn dust in alc. alk. sol. → 2 : 4-dimethylbenzoic acid.

Bösesen, *Rec. trav. chim.*, 1907, **26**, 285.

Cohen, *Rec. trav. chim.*, 1919, **38**, 119.

**2 : 5 : 2' : 5' - Tetramethylbenzophenone.**

Viscous liq. B.p. 325-7°. Prolonged boiling → 1 : 4 : 6-trimethylantracene.

Elbs, *J. prakt. Chem.*, 1887, **35**, 481.

**3 : 4 : 3' : 4' - Tetramethylbenzophenone.**

Cryst. from EtOH. M.p. 140°. Sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ .

*Oxime*: cryst. from EtOH. M.p. 147°.

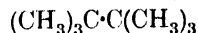
*Phenylhydrazone*: yellow micro-cryst. M.p. 130°.

Bistrzycki, Reintke, *Ber.*, 1905, **38**, 844.

**2 : 3 : 5 : 6 - Tetramethylbenzoquinone.**

See Duroquinone.

**2 : 2 : 3 : 3 - Tetramethylbutane** (*Hexamethylethane, di-tert.-butyl*)



$C_8H_{18}$

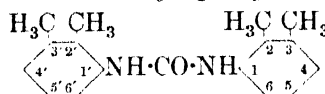
MW, 114

Cryst. from  $Et_2O$ . M.p. 104° (100.7-101.4°). B.p. 106-7°. Volatile.

Flood, Calingaert, *J. Am. Chem. Soc.*, 1934, **56**, 1211.

Whitmore, Stehman, Herndon, *J. Am. Chem. Soc.*, 1933, **55**, 3807.

**2 : 3 : 2' : 3' - Tetramethylcarbanilide**  
(*2 : 3 : 2' : 3' - Tetramethyldiphenylurea*)



$C_{17}H_{20}ON_2$

MW, 268

Cryst. from EtOH. M.p. 242°.

Mazourewitch, *Bull. soc. chim.*, 1924, **35**, 1185.

**2 : 4 : 2' : 4' - Tetramethylcarbanilide**  
(*2 : 4 : 2' : 4' - Tetramethyldiphenylurea*).

Needles from AcOH. M.p. 263-5°.

Thomson, Wilson, *J. Chem. Soc.*, 1933, 1263.

**2 : 5 : 2' : 5' - Tetramethylcarbanilide**  
(*2 : 5 : 2' : 5' - Tetramethyldiphenylurea*).

Needles from AcOH. M.p. 285° (sealed tube).

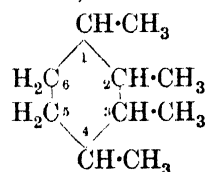
See previous reference.

**3 : 4 : 3' : 4' - Tetramethylcarbanilide**  
(*3 : 4 : 3' : 4' - Tetramethyldiphenylurea*).

M.p. 236°.

Mailhe, *Compt. rend.*, 1923, **176**, 903.

**1 : 2 : 3 : 4 - Tetramethylcyclohexane**  
(*Hexahydroprehnitene*)



$C_{10}H_{20}$

MW, 140

B.p. 84°/5 mm.  $D_4^{20}$  0.8219.  $n_D^{20}$  1.4531.

Mitchell, Marvel, *J. Am. Chem. Soc.*, 1933, **55**, 4278.

**1 : 2 : 3 : 5 - Tetramethylcyclohexane**  
(*Hexahydroisodurene*).

*Cis*:

B.p. 168-70°/762 mm.  $D_4^{20}$  0.8166.  $n_D^{20}$  1.44847.

*Trans* :

B.p. 162-4°/765 mm.  $D_4^{20}$  0.8140.  $n_{D_{16}}^{20}$  1.44657.

Eisenlohr, *Chem. Abstracts*, 1926, 20, 171.

**1 : 2 : 4 : 5-Tetramethylcyclohexane**  
(*Hexahydrodurene*).

The following compounds have been described :

(i) B.p. 172-4°/730 mm.  $D_4^{20}$  0.7759.  $n_D^{20}$  1.4205.

Pictet, Ramseyer, Kaiser, *Ann. chim.*, 1918, 10, 297.

(ii) B.p. 169-170.5°/711 mm.  $D_4^{20}$  0.811.  $n_D^{20}$  1.4451.

Willstätter, Hatt, *Ber.*, 1912, 45, 1473.

(iii) B.p. 160.5-161.5°.  $D_4^{13.1}$  0.7910.  $n_D^{13.1}$  1.437.

Auwers, *Ann.*, 1920, 420, 108.

(iv)

*Cis* :

B.p. 171°/755 mm.  $D_4^{20}$  0.8122.  $n_{D_{16}}^{20}$  1.44647.

*Trans* :

B.p. 166-8°.  $D_4^{20}$  1.8100.  $n_{D_{16}}^{20}$  1.44446.

Eisenlohr, *Chem. Abstracts*, 1926, 20, 171.

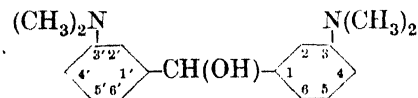
**1 : 2 : 2 : 3-Tetramethylcyclopentane-1-carboxylic Acid.**

See Campholic Acid.

**1 : 2 : 3 : 3-Tetramethylcyclopentene.**

See Campholene.

**3 : 3'-Tetramethyldiaminobenzhydrol**



$C_{17}H_{22}ON_2$  MW, 270

Prisms from  $Et_2O$ . M.p. 72-3°. Sols. in acids are colourless.

Baeyer, *Ann.*, 1907, 354, 194.

**3 : 4'-Tetramethyldiaminobenzhydrol.**

Needles from EtOH. M.p. 100-1°. Sol. hot AcOH with yellowish-green col.

Baeyer, *Ann.*, 1907, 354, 191.

**4 : 4' - Tetramethyldiaminobenzhydrol**  
(*Michler's Hydrol*).

Green leaflets from  $C_6H_6$ . M.p. 98° (102-3°). Sol.  $Et_2O$ , AcOH,  $C_6H_6$ , hot EtOH. Insol.  $H_2O$ . 1 mol. Br in cold  $\rightarrow$  *p*-dimethylaminobenzaldehyde + *p*-bromodimethylaniline. Gives blue col. with  $H\cdot CHO$ ,  $CH_3\cdot CHO$ , chloral,  $Me_2CO$ ,  $C_6H_5\cdot CHO$ , and acetophenone. Intermediate for basic dyes.

*Me ether* :  $C_{18}H_{24}ON_2$ . MW, 284. Cryst from ligroin. M.p. 71-2°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. ligroin.

*Benzyl ether* : plates from ligroin. M.p. 102-3°.

*Dimethiodide* : leaflets from EtOH. M.p. 195°.

$B_3C_6H_3(NO_2)_3$ -1 : 3 : 5 : black needles. M.p. 75-5°.

Bogert, Ruderman, *J. Am. Chem. Soc.*, 1922, 44, 2616.

Cohen, *Rec. trav. chim.*, 1919, 38, 121.

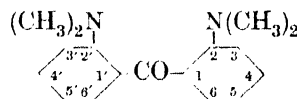
Badische, D.R.P., 27,032.

Bielecki, Koleniew, *Chem. Zentr.*, 1908, II, 877.

Möhlau, Heinze, *Ber.*, 1902, 35, 360.

National Aniline & Chemical Co., U.S.P., 1,942,820, (*Chem. Zentr.*, 1934, I, 2826).

**2 : 2'-Tetramethyldiaminobenzophenone**



$C_{17}H_{20}ON_2$

MW, 268

Yellow prisms or needles from EtOH. M.p. 122° (117-18°). Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ . Volatile in steam. Does not form oxime or phenylhydrazone.

*Picrate* : yellow leaflets from EtOH. Decomp. at 160-2°.

Baeyer, *Ber.*, 1905, 38, 2764.

Bertram, *J. prakt. Chem.*, 1902, 65, 340.

**3 : 3'-Tetramethyldiaminobenzophenone.**

Yellow prisms from EtOH.Aq. M.p. 59-60°. Sol. most org. solvents. Sols. in acids are colourless.

Baeyer, *Ann.*, 1907, 354, 193.

**3 : 4'-Tetramethyldiaminobenzophenone.**

Brownish-yellow cryst. from EtOH or  $Et_2O$ . M.p. 77-78.5°.

*Hydrochloride* : needles from EtOH. M.p. 278-80° decomp. Decomp. by  $H_2O$ .

Baeyer, *Ann.*, 1907, 354, 190.

**4 : 4'-Tetramethyldiaminobenzophenone**  
(*Michler's Ketone*).

Leaflets from EtOH. M.p. 179° (172-172.5°). B.p. above 360° decomp. Sol. warm  $C_6H_6$ . Mod. sol. EtOH. Very spar. sol.  $Et_2O$ . Insol.  $H_2O$ . NaHg in EtOH  $\rightarrow$  4 : 4'-tetramethyldiaminobenzhydrol. Intermediate for basic dyes.

*Oxime* : cryst. from EtOH. M.p. 233°.

**2 : 2'-Tetramethyldiaminodiphenyl**

726

*Phenylhydrazone*: needles from  $C_6H_6$ -EtOH. M.p. 174-5°.

*Di-Me acetal*: pale yellow needles from MeOH. M.p. 130°.

*Di-Et acetal*: m.p. 118°.

*Dimethobromide*: yellowish plates +  $2H_2O$  from EtOH.Aq. M.p. anhyd. 168°.

*Dimethiodide*: yellowish leaflets from EtOH. M.p. 105°. At 150° decomp. into its components.

*Hydrazone*: yellow needles from EtOH. M.p. 150°.

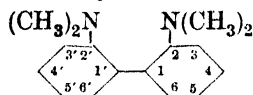
*Azine*: brownish-red prisms from xylene. M.p. 253°.

*Picrate*: purple-red prisms. M.p. 156-7°.

Michler, Moro, *Ber.*, 1879, 12, 1168.

Michler, *Ber.*, 1876, 9, 716.

Fehrmann, *Ber.*, 1887, 20, 2845.

**2 : 2'-Tetramethyldiaminodiphenyl**

$C_{16}H_{20}N_2$  MW, 240

Plates from pet. ether. M.p. 72-3°.

*Hydriodide*: m.p. 256-7°.

*Methiodide*: prisms from  $H_2O$ . M.p. 190-2°.

Shaw, Turner, *J. Chem. Soc.*, 1933, 139.

**3 : 3'-Tetramethyldiaminodiphenyl.**

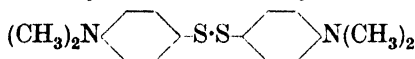
Needles from EtOH. M.p. 126-8°.

Dutt, *J. Chem. Soc.*, 1926, 1181.

**4 : 4'-Tetramethyldiaminodiphenyl.**

See *N : N'*-Tetramethylbenzidine.

**Tetramethyldiaminodiphenyl disulphide**  
(*Dithiodimethylaniline*, *tetramethyldithioaniline*)



$C_{16}H_{20}N_2S_2$  MW, 304

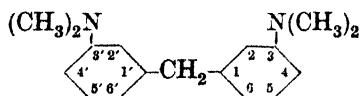
Yellow needles from EtOH. M.p. 118°. Sol.  $CS_2$ . Spar. sol. EtOH,  $Et_2O$ , ligroin, hot  $C_6H_6$ . Insol.  $H_2O$ .

Söderbäck, *Ann.*, 1919, 419, 276.

Merz, Weith, *Ber.*, 1886, 19, 1571.

**Tetramethyldiaminodiphenyl Ether.**

See under *N*-Dimethyl-*p*-aminophenol.

**3 : 3' - Tetramethyldiaminodiphenyl - methane**

$C_{17}H_{22}N_2$  MW, 254

**4 : 4'-Tetramethyldiaminotriphenyl-methane**

Yellowish oil with green fluor.

*Dimethiodide*: needles from  $H_2O$ . M.p. 165°.

Scholl, *Monatsh.*, 1918, 39, 236.

**4 : 4' - Tetramethyldiaminodiphenyl - methane (Methane Base).**

Leaflets or plates from EtOH or ligroin. M.p. 91°. B.p. 390°. Non-volatile in steam. Sol.  $Et_2O$ ,  $C_6H_6$ , hot EtOH. Sol. acids. Ox. → Michler's Hydrol → Michler's Ketone.

$B_2C_6H_4(NO_2)_2$ -1 : 3 : red cryst. M.p. 76°.

$B_2C_6H_3(NO_2)_3$ -1 : 3 : 5 : violet needles. M.p. 114°.

*Monopicrate*: yellow leaflets. M.p. 185°.

*Dipicrate*: m.p. 178°.

*Dimethiodide*: yellow leaflets. Becomes green at 193° and melts at 214° decomp.

*N : N'*-Dioxide: needles +  $2H_2O$  from EtOH- $Et_2O$ . M.p. 147°, anhyd. 156°. Very sol.  $H_2O$ , EtOH. Reacts alkaline in solution.  $B_2HCl$ : m.p. 165.5-166° decomp. *Dipicrate*: yellow needles from EtOH. M.p. 150.5-151° decomp.

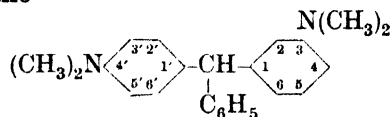
Cohn, *Chem.-Ztg.*, 1900, 24, 564.

Fischl, *Monatsh.*, 1914, 35, 531.

Votoček, Krauz, *Ber.*, 1909, 42, 1604.

Nathansohn, Müller, *Ber.*, 1889, 22, 1882.

Höchst, D.R.P., 107,718, (*Chem. Zentr.*, 1900, I, 1110).

**3 : 4' - Tetramethyldiaminotriphenyl - methane**

$C_{23}H_{26}N_2$  MW, 330

Cryst. from EtOH. M.p. 83-4°. Mod. sol.  $Et_2O$ , hot EtOH.

Baeyer, *Ann.*, 1907, 354, 197.

**4 : 4' - Tetramethyldiaminotriphenyl - methane (Leuco-Malachite Green).**

Cryst. in three forms. (i) Needles from  $C_6H_6$ . M.p. 102°. (ii) Plates from EtOH. M.p. 93-4°. (iii) Cryst. of lower indefinite m.p. Distills undecomp. in small quantities. Sol.  $Et_2O$ ,  $C_6H_6$ , toluene. Mod. sol. EtOH. Spar. sol. ligroin. Insol.  $H_2O$ .

$B_2C_6H_3(NO_2)_3$ -1 : 3 : 5 : m.p. 88.5-89°.

*Dimethiodide*: plates or needles from  $H_2O$ . M.p. 231° (218-22°) decomp. into components.

*N : N'*-Dioxide: needles + 2 or  $4H_2O$  from  $CHCl_3$ . M.p. 131.5-132.5°, anhyd. 188-9°.

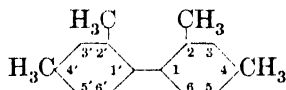
Very sol.  $\text{H}_2\text{O}$ , EtOH. Very spar. sol.  $\text{Et}_2\text{O}$ , ligroin. Reacts alkaline.

Nencki, *Monatsh.*, 1888, 9, 1148.

Fischer, *Ann.*, 1881, 206, 122, 136.

Fischer, Fischer, Lehmann, *Ber.*, 1879, 12, 798.

### 2 : 4 : 2' : 4'-Tetramethyldiphenyl



$\text{C}_{16}\text{H}_{18}$

MW, 210

Cryst. from EtOH. M.p.  $41^\circ$ . B.p.  $288^\circ/722$  mm. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , warm EtOH.

Scholl, Liese, Michelson, Grunewald, *Ber.*, 1910, 43, 513.

### 2 : 5 : 2' : 5'-Tetramethyldiphenyl.

Cryst. from EtOH. M.p.  $50^\circ$ . B.p.  $284^\circ/732$  mm. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

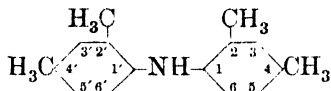
Ullmann, Meyer, *Ann.*, 1904, 332, 47.

### 3 : 4 : 3' : 4'-Tetramethyldiphenyl.

Yellowish needles from EtOH. M.p.  $76-7^\circ$ . Sol.  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ , AcOEt,  $\text{C}_6\text{H}_6$ . Volatile in steam.

Crossley, Hampshire, *J. Chem. Soc.*, 1911, 99, 726.

### 2 : 4 : 2' : 4'-Tetramethyldiphenylamine



$\text{C}_{16}\text{H}_{19}\text{N}$

MW, 225

Cryst. M.p.  $58-58.5^\circ$ . B.p.  $305-10^\circ$ .

$\text{B}_2\text{HCl}$ : m.p.  $166^\circ$  decomp.

Müller, *Ber.*, 1887, 20, 1042.

Bamberger, Brun, *Helv. Chim. Acta*, 1924, 7, 118.

### 3 : 4 : 3' : 4'-Tetramethyldiphenylamine.

Viscous oil. B.p.  $340-5^\circ$  decomp. ( $330-45^\circ$ ). Volatile in steam.

Müller, *Ber.*, 1887, 20, 1041.

### Tetramethyldiphenylurea.

See Tetramethylcarbanilide.

### 4 : 8 : 12 : 16-Tetramethyleicosane.

See Bixane.

### Tetramethylene.

See Cyclobutane.

### Tetramethylene bromide.

See 1 : 4-Dibromobutane.

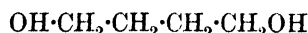
### Tetramethylene chloride.

See 1 : 4-Dichlorobutane.

### Tetramethylenediamine.

See Putrescine.

### Tetramethylene Glycol (1 : 4-Dihydroxybutane, butandiol-1 : 4)



$\text{C}_4\text{H}_{10}\text{O}_2$

MW, 90

Viscous liq. Solidifies in freezing mixture to colourless needles, m.p.  $16^\circ$ . B.p.  $230^\circ$ ,  $120^\circ/10$  mm. Sol.  $\text{H}_2\text{O}$ , EtOH. Spar. sol.  $\text{Et}_2\text{O}$ .  $D_4^{20}$  1.020. Dil.  $\text{HNO}_3 \rightarrow$  succinic acid. Bitter taste. Ppd. from aq. sol. by  $\text{K}_2\text{CO}_3$ .

*Di-Me ether*:  $\text{C}_6\text{H}_{14}\text{O}_2$ . MW, 118. B.p.  $133^\circ$ .  $D_4^{15}$  0.8664.  $n_D^{15}$  1.4031.

*Me-Et ether*:  $\text{C}_7\text{H}_{16}\text{O}_2$ . MW, 132. B.p.  $145-6^\circ/757$  mm.  $D_4^{15}$  0.8484.  $n_D^{15}$  1.4012. Sol. EtOH,  $\text{Et}_2\text{O}$ .

*Di-Et ether*:  $\text{C}_8\text{H}_{18}\text{O}_2$ . MW, 146. B.p.  $155-7^\circ/730$  mm.,  $59-60^\circ/18$  mm.  $D_4^{20}$  0.8455.  $n_D^{20}$  1.40610.

*Dipropyl ether*:  $\text{C}_{10}\text{H}_{22}\text{O}_2$ . MW, 174. B.p.  $94-5^\circ/20$  mm.  $D_4^{20}$  0.8409.  $n_D^{20}$  1.41368.

*Di-isopropyl ether*: b.p.  $77-8^\circ/18$  mm.  $D_4^{20}$  0.8310.  $n_D^{20}$  1.40954.

*Diacetyl*: m.p.  $12^\circ$ . B.p.  $230^\circ$ ,  $124^\circ/20$  mm.  $D_4^{15}$  1.0479.  $n_D^{15}$  1.4251.

*Carbonate*: m.p.  $59^\circ$ .

*Succinate*: monomer, m.p.  $42^\circ$ . B.p.  $95-6^\circ/2$  mm.  $D_4^{60}$  1.1732.  $n_D^{20}$  1.4567. Dimer: m.p.  $121^\circ$ .

*Azelate*: monomer, m.p.  $9^\circ$ . B.p.  $123-4^\circ/2$  mm.

*Sebacate*: monomer, m.p.  $6^\circ$ . B.p.  $136-8^\circ/2$  mm.

*Dibenzoyl*: prisms or needles from pet. ether. M.p.  $57.5^\circ$  ( $53^\circ$ ).

*Di-phenylurethane*: cryst. from  $\text{CHCl}_3$ . M.p.  $183-183.5^\circ$ .

*Di-1-naphthylurethane*: needles from butyl alcohol. M.p.  $198^\circ$ .

Wojcik, Adkins, *J. Am. Chem. Soc.*, 1934, 56, 2423.

Tallman, *ibid.*, 128.

Müller, Clostermeyer, *Monatsh.*, 1928, 49, 28.

Bennett, Heathcoat, *J. Chem. Soc.*, 1929, 271.

Bösesken, *Rec. trav. chim.*, 1915, 34, 100.

### Tetramethyleneimine.

See Pyrrolidine.

### Tetramethylene iodide.

See 1 : 4-Di-iodobutane.

### Tetramethylene oxide.

See Tetrahydrofuran.

### Tetramethylene sulphide.

See Tetrahydrothiophene.



**Tetramethylene sulphone.**

See under Tetrahydrothiophene.

**Tetramethylethane.**

See Dimethylbutane.

**Tetramethylethylene.**

See 2 : 3-Dimethylbutylene-2.

**Tetramethylethylene glycol.**

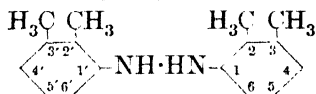
See Pinacol.

**2 : 6 : 11 : 15-Tetramethylhexadecane.**

See Crocetane.

**Tetramethylhydricrylic Acid.**

See 2-Hydroxy-1 : 1-dimethylisovaleric Acid.

**2 : 3 : 2' : 3'-Tetramethylhydrazobenzene**  
(3 : 3'-Hydrazo-o-xylene) $C_{16}H_{20}N_2$ 

MW, 240

Colourless needles from EtOH. M.p. 149–50° (139–40°). Sol. most org. solvents. Oxidises easily in air.

Bamberger, *Ber.*, 1926, 59, 428.**2 : 4 : 2' : 4'-Tetramethylhydrazobenzene**  
(4 : 4'-Hydrazo-m-xylene).

Needles from EtOH. M.p. 125–6° (120–2°).

See previous reference.

**2 : 5 : 2' : 5'-Tetramethylhydrazobenzene**  
(2 : 2'-Hydrazo-p-xylene).

Needles from EtOH. M.p. 145°.

Nölting, Stricker, *Ber.*, 1888, 21, 3143.**3 : 4 : 3' : 4'-Tetramethylhydrazobenzene**  
(4 : 4'-Hydrazo-o-xylene).

Needles from EtOH. M.p. 113–14° (106–7°).

Bamberger, *Ber.*, 1926, 59, 428.**3 : 5 : 3' : 5'-Tetramethylhydrazobenzene**  
(5 : 5'-Hydrazo-m-xylene).

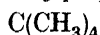
Needles from EtOH. M.p. 124–5°. Oxidises readily in air.

Nölting, Stricker, *Ber.*, 1888, 21, 3142.**Tetramethylhydroquinone.**

See Durohydroquinone.

**Tetramethylhydric Acid.**

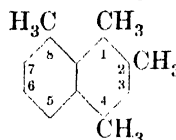
See Deoxyamalic Acid.

**Tetramethylmethane** (2-Methylisobutane, neopentane, 2 : 2-dimethylpropane) $C_5H_{12}$ 

MW, 72

F.p. – 19.5°. B.p. 9.4°/760 mm.  $D_4^{20}$  0.613.  $n_D^{20}$  1.3513.Whitmore, Fleming, *J. Am. Chem. Soc.*, 1933, 55, 3803.**Tetramethylmethane - tetracarboxylic Acid.**

See Methane-tetracetic Acid.

**1 : 2 : 4 : 8-Tetramethylnaphthalene** $C_{14}H_{16}$ 

MW, 184

Oil. B.p. 150°/10 mm.

Picrate : red needles. M.p. 145.5°.

Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, 16, 324.**1 : 2 : 5 : 6-Tetramethylnaphthalene.**

Cryst. from EtOH. M.p. 118°. B.p. 150–60°/10 mm.

Picrate : red needles. M.p. 156–7° (154–154.5°).

Styphnate : cryst. from  $C_6H_6$ . M.p. 166° (162°). $C_{14}H_{16}, C_6H_3(NO_2)_3-1 : 3 : 5 :$  orange-red needles from MeOH. M.p. 180–180.5° (178.5–179°).Brunner, Hofer, Stein, *Monatsh.*, 1933, 63, 96.Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, 16, 320.**1 : 2 : 5 : 7-Tetramethylnaphthalene.**

B.p. 155–8°/12 mm.

Picrate : orange-yellow needles from MeOH. M.p. 144–5°.

Styphnate : orange-yellow needles from MeOH. M.p. 144–5°.

 $C_{14}H_{16}, C_6H_3(NO_2)_3-1 : 3 : 5 :$  pale yellow needles from EtOH. M.p. 167–8°.Hosking, Brandt, *Ber.*, 1935, 68, 289.Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, 16, 323.**1 : 2 : 5 : 8-Tetramethylnaphthalene.**

Oil. B.p. 150°/9 mm.

Picrate : red cryst. M.p. 137–8°.

 $C_{14}H_{16}, C_6H_3(NO_2)_3-1 : 3 : 5 :$  orange - yellow needles. M.p. 158–9°.Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, 16, 322.**1 : 2 : 6 : 8-Tetramethylnaphthalene.**

Oil. B.p. 166–8°/15 mm.

Picrate : orange-red needles. M.p. 133.5–134°.

Styphnate : orange needles. M.p. 135–6°.

Ruzicka, Ehmann, Mörgeli, *Helv. Chim. Acta*, 1933, 16, 321.

**2 : 2 : 4 : 4-Tetramethylpentanol-3.**

See Di-tert.-butylcarbinol.

**2 : 2 : 4 : 4-Tetramethylpentanone-3.**

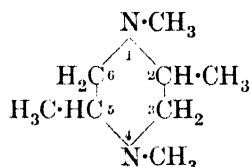
See Pivalone.

**2 : 3 : 4 : 5-Tetramethylphenol.**

See Prehnitenol.

**Tetramethylphenylenediamine.**

See under Phenylenediamine.

**1 : 2 : 4 : 5-Tetramethylpiperazine** $C_8H_{18}N_2$ 

MW, 142

Syrup.

 $B,2HCl$ : m.p. 257° decomp. $B,HI$ : cryst. M.p. 178° decomp. $Dimethiodide$ : cryst. from MeOH. M.p. 250°. $Dihydroxymethylate$ : cryst. from  $Et_2O$ -pet. ether. M.p. 224°.Abderhalden, Haas, *Z. physiol. Chem.*, 1925, **149**, 94.**1 : 2 : 4 : 6-Tetramethylpiperazine.**Colourless liq. B.p. 163-4°. Misc. with  $H_2O$  and most org. solvents in all proportions. $B,2H_2P_2Cl_6$ : orange needles +  $2H_2O$  from  $H_2O$ . Darkens at 270°. Decomp. at 275°. $Monomethiodide$ : needles from  $Et_2O$ -EtOH. M.p. 227°. $d$ -Camphor- $\beta$ -sulphonate: needles or prisms from EtOH- $Me_2CO$ . M.p. 223°.  $[\alpha]_{5461} + 21.6^\circ$  in  $H_2O$ . $d$ - $\alpha$ -Bromocamphor- $\beta$ -sulphonate: cryst. from  $Me_2CO$ . M.p. 175°.  $[\alpha]_{5461} + 97.4^\circ$  in  $H_2O$ . $d$ - $\alpha$ -Bromocamphor- $\pi$ -sulphonate: needles from  $Me_2CO$ . M.p. 249°.  $[\alpha]_{5461} + 88.1^\circ$  in  $H_2O$ . $Picrate$ : yellow cryst. Decomp. at 280°.Pope, Read, *J. Chem. Soc.*, 1914, **105**, 224.**2 : 2 : 5 : 5-Tetramethylpiperazine.** $Dinitroso deriv.$ : cryst. from AcOH. M.p. 208-10°.Conant, Aston, *J. Am. Chem. Soc.*, 1928, **50**, 2788.**2 : 3 : 5 : 6-Tetramethylpiperazine.**

Five optically inactive stereoisomeric forms are possible.

I.

Colourless needles. M.p. 45°. B.p. 177-8°. Readily sol.  $H_2O$ ,  $CHCl_3$ . $Dihydrate$ : needles from  $Me_2CO$ . M.p. 84-5°. Very sol.  $H_2O \rightarrow$  alk. sol. $B,2HCl$ : prisms from EtOH.Aq. Does not melt below 300°. $Dinitroso deriv.$ : needles from EtOH. M.p. 157°. $Dibenzoyl$ : plates from EtOH. M.p. 247-8°. $Di$ -p-toluenesulphonyl: cryst. from Py. M.p. 308-9°.

II.

Liq. B.p. 183°. Very sol.  $H_2O$ . $B,2HCl$ : prisms from EtOH. Does not melt below 300°. $Dinitroso deriv.$ : yellow prisms from EtOH. M.p. 101-2°. $Dibenzoyl$ : prisms from  $Me_2CO$ -pet. ether. M.p. 175-6°. $Mono$ -p-toluenesulphonyl: needles from EtOH.

M.p. 81-2°.

 $Di$ -p-toluenesulphonyl: plates from Py.Aq. M.p. 222°. $d$ -Camphor-10-sulphonate: m.p. 89-90°.  $[\alpha]_{5461}^{15} + 35.7^\circ$  in  $CHCl_3$ .

III.

Colourless cryst. M.p. 67-8°. B.p. 195-6°.

 $B,2HCl$ : plates +  $1H_2O$  from EtOH.Aq. Does not melt below 300°. $Dinitroso deriv.$ : yellow prisms from EtOH. M.p. 173-4°. $Monobenzoyl$ : prisms from pet. ether. M.p. 85°. $Dibenzoyl$ : prisms from EtOH. M.p. 163-4°. $Mono$ -p-toluenesulphonyl: plates from EtOH.Aq. or pet. ether. M.p. 138-9°. $N$ -Me:  $C_9H_{20}N_2$ . MW, 156. M.p. 4-5°. B.p. 201-2°. Very hygroscopic.  $Monohydrate$ :prisms from  $Me_2CO$ . M.p. 73-4°.  $B,HI$ : prisms from EtOH or  $H_2O$ . M.p. 161-2°. $B,2HCl$ : needles from EtOH.Aq.- $Me_2CO$ . M.p. about 300°. $B,2HI$ : prisms from EtOH. M.p. 240° decomp.  $Nitroso deriv.$ : m.p. 24-5°. B.p. 155-7°/15 mm. $N$ : $N$ -Di-Me:  $C_{10}H_{22}N_2$ . MW, 170. B.p. 211-12°.  $Monomethiodide$ : needles from MeOH- $Me_2CO$ . M.p. 272-4°.

IV.

Hydrated needles from  $H_2O$ . M.p. 53-5°.  $B,2HCl$ : prisms from  $H_2O$ . Does not melt below 300°. $Dinitroso deriv.$ : yellow prisms from  $Me_2CO$ . M.p. 189-90°.

V.

 $B,2HCl$ : prisms from EtOH.Aq.

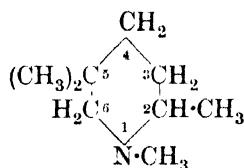
*Dinitroso deriv.*: yellow prisms from EtOH or Me<sub>2</sub>CO. M.p. 116–17°.

*Dibenzoyl*: needles from Me<sub>2</sub>CO or pet. ether. M.p. 146–7°.

Stoehr, Brandes, *J. prakt. Chem.*, 1897, 55, 74.

Kipping, *J. Chem. Soc.*, 1937, 368; 1932, 1336; 1931, 1160; 1929, 2889.

## 1 : 2 : 5 : 5-Tetramethylpiperidine

C<sub>9</sub>H<sub>19</sub>N

MW, 141

Liq. B.p. 147–8°.

*B, HCl*: m.p. about 221° decomp.

Mannich, Lesse, *Arch. Pharm.*, 1933, 271, 92, (*Chem. Abstracts*, 1934, 28, 477).

## 2 : 2 : 6 : 6-Tetramethylpiperidine.

Liq. B.p. 155.5–156.5°. Volatile in steam. D<sub>4</sub><sup>15.2</sup> 0.8367.

*Hydrate*: needles. M.p. 28°.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: cryst. M.p. 270°.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: prisms from H<sub>2</sub>O. M.p. 174°.

*B<sub>2</sub>HNO<sub>2</sub>*: cryst. from CHCl<sub>3</sub>. Decomp. at 270°.

*B<sub>2</sub>HAuCl<sub>4</sub>*: yellow needles from H<sub>2</sub>O. M.p. 165°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-red prisms from H<sub>2</sub>O. M.p. 262°.

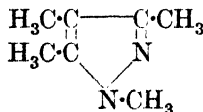
*N-Benzoyl*: plates from EtOH. M.p. 41–2°.

Franchimont, Friedmann, *Rec. trav. chim.*, 1905, 24, 404.

## 2 : 2 : 6 : 6-Tetramethyl-γ-piperidone.

See Triacetoneamine.

## 1 : 3 : 4 : 5-Tetramethylpyrazole

C<sub>7</sub>H<sub>12</sub>N<sub>2</sub>

MW, 124

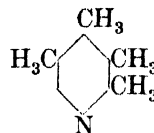
Liq. with unpleasant odour. Cryst. on cooling. B.p. 190–3°. Misc. in all proportions with H<sub>2</sub>O, Et<sub>2</sub>O, EtOH.

*Picrate*: m.p. 176–8°.

Rojahn, Kühling, *Arch. Pharm.*, 1926, 264, 337, (*Chem. Abstracts*, 1926, 20, 2856).

Knorr, Oettinger, *Ann.*, 1894, 279, 246.

## 2 : 3 : 4 : 5-Tetramethylpyridine

C<sub>9</sub>H<sub>13</sub>N

MW, 135

Liq. B.p. 232–4°. Mod. misc. with H<sub>2</sub>O. KMnO<sub>4</sub> → pyridine-2 : 3 : 4 : 5-tetracarboxylic acid.

*B<sub>2</sub>HAuCl<sub>4</sub>*: yellow needles from H<sub>2</sub>O. M.p. 216–18°.

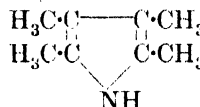
*B<sub>2</sub>(HgCl<sub>2</sub>)<sub>2</sub>HCl*: needles from H<sub>2</sub>O. M.p. 159°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: needles from H<sub>2</sub>O. M.p. 209–10° decomp.

*Picrate*: needles from H<sub>2</sub>O. M.p. 170–2°.

Ahrens, *Ber.*, 1895, 28, 796.

## 2 : 3 : 4 : 5-Tetramethylpyrrole

C<sub>8</sub>H<sub>13</sub>N

MW, 123

Leaflets from EtOH.Aq. or pet. ether. M.p. 111°. B.p. 130°/7 mm. Misc. with most org. solvents. Volatile in steam with part. decomp.

*Picrate*: yellow cryst. from C<sub>6</sub>H<sub>6</sub>, EtOH, or Et<sub>2</sub>O. M.p. 130°.

*Styphnate*: reddish-brown needles. M.p. 159°.

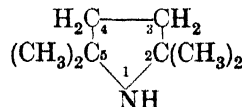
Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, 58, 714.

Neitzescu, Solomonica, *Ber.*, 1931, 64, 1928.

Fischer, Zerweck, *Ber.*, 1923, 56, 525.

Fischer, Bartholomäus, *Z. physiol. Chem.*, 1913, 87, 269; 1913, 83, 65.

## 2 : 2 : 5 : 5-Tetramethylpyrrolidine

C<sub>8</sub>H<sub>17</sub>N

MW, 127

Pale rose-coloured oil. B.p. 108°. With pine splint → red col. Isatin + H<sub>2</sub>SO<sub>4</sub> → greenish-blue col.

*Benzoyl*: cryst. M.p. 67.5–68°. Sol. cold pet. ether.

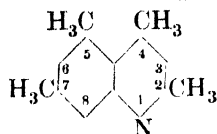
Pace, *Chem. Abstracts*, 1928, 22, 3890.

Konowalow, Wojnitsch - Sjanoshenski, *Chem. Zentr.*, 1905, II, 830.

**2 : 3 : 4 : 5-Tetramethylpyrrolidine.**

*N-Et*:  $C_{10}H_{21}N$ . MW, 255. B.p. 163–5°.  $D_{15}^{20}$  0.8064.  $n_D^{20}$  1.4343.

Signaigo, Adkins, *J. Am. Chem. Soc.*, 1936, **58**, 715.

**2 : 4 : 5 : 7-Tetramethylquinoline**

$C_{13}H_{15}N$  MW, 185

Cryst. from pet. ether. M.p. 59°.

Mikeska, Adams, *J. Am. Chem. Soc.*, 1920, **42**, 2394.

**2 : 4 : 5 : 8-Tetramethylquinoline.**

Pale yellow cryst. M.p. 48°. B.p. 168–72°/12 mm. Acid sol. → bluish-violet fluorescence.

*B.HCl*: sinters at 243°. M.p. 254°.

*Picrate*: needles from EtOH. M.p. 161°.

Braun, Gmelin, Petzold, *Ber.*, 1924, **57**, 389.

**2 : 4 : 6 : 8-Tetramethylquinoline.**

Plates from Et<sub>2</sub>O or pet. ether. M.p. 86°. B.p. 284–5°. Very sol. Et<sub>2</sub>O.

*B.H<sub>2</sub>SO<sub>4</sub>*: needles from EtOH–Et<sub>2</sub>O. M.p. 243° decomp.

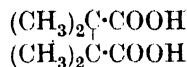
Mikeska, Adams, *J. Am. Chem. Soc.*, 1920, **42**, 2394.

**2 : 5 : 6 : 8-Tetramethylquinoline.**

Oil. F.p. 20° to cryst. mass. B.p. 297–300°. Very sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Doebner, Miller, *Ber.*, 1884, **17**, 1710.

**Tetramethylsuccinic Acid** (2 : 3-Dimethylbutane-2 : 3-dicarboxylic acid)



$C_8H_{14}O_4$  MW, 174

Cryst. M.p. 200°. Sol. 201 parts H<sub>2</sub>O at 13.5°. Very sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>. Insol. ligroin.  $k = 3.14 \times 10^{-4}$  at 25°. Heat above m.p. → anhydride.

*Mono-Me ester*:  $C_9H_{16}O_4$ . MW, 188. Prisms from pet. ether. M.p. 68°.  $k = 1.22 \times 10^{-5}$  at 25°.

*Di-Me ester*:  $C_{10}H_{18}O_4$ . MW, 202. Prisms from ligroin. M.p. 31°. Very sol. most org. solvents.

*Mono-Et ester*:  $C_{10}H_{18}O_4$ . MW, 202. Viscous oil. Dist. → anhydride + C<sub>2</sub>H<sub>5</sub>OH.

*Di-Et ester*:  $C_{12}H_{22}O_4$ . MW, 230. B.p. 219°/13 mm.  $D_{15}^{20}$  0.995.  $n_D^{20}$  1.436.

*Imide*:  $C_8H_{13}O_2N$ . MW, 155. Needles from C<sub>6</sub>H<sub>6</sub>–ligroin. M.p. 187°. Spar. sol. ligroin. Sublimes.

*p-Tolylimide*: needles from EtOH.Aq. M.p. 90°.

*2-Naphthylimide*: needles from EtOH.Aq. M.p. 152°.

*Anhydride*:  $C_8H_{12}O_3$ . MW, 156. Needles from ligroin. M.p. 147°. B.p. 230–5°. Spar. sol. H<sub>2</sub>O, ligroin. Sublimes.

*Di-nitrile*:  $C_8H_{12}N_2$ . MW, 136. Plates or prisms from EtOH.Aq. M.p. 169°.

*Mono-anilide*: cryst. from Et<sub>2</sub>O–pet. ether. M.p. 91.5–92°.

*Anil*: needles from EtOH.Aq. M.p. 88°.

*Mono-p-toluidide*: cryst. M.p. 116–17°.

*p-Tolil*: cryst. M.p. 91.5°.

Auwers, Ungemach, *Ber.*, 1935, **68**, 351.

Auwers, Ottens, *Ber.*, 1924, **57**, 440.

Auwers, Meyer, *Ber.*, 1890, **23**, 297.

**1 : 1 : 2 : 6 - Tetramethyl - 1 : 2 : 3 : 4 - tetrahydronaphthalene.**

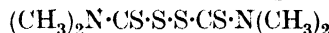
See Irene.

**1 : 1 : 2 : 6-Tetramethyltetralin.**

See Irene.

**Tetramethylthiourea.**

See under Thiourea.

**Tetramethylthiuram disulphide**

$C_6H_{12}N_2S_4$  MW, 240

White cryst. from CHCl<sub>3</sub>–EtOH. M.p. 146°. Very sol. CHCl<sub>3</sub>. Spar. sol. EtOH, Et<sub>2</sub>O. KCN → tetramethylthiuram sulphide. Used as accelerator in vulcanisation of rubber.

Cummings, Simmons, *Ind. Eng. Chem.*, 1928, **20**, 1173, (*Brit. Chem. Abstracts*, 1929, B, 28).

Romani, *Chem. Abstracts*, 1922, **16**, 854.

v. Braun, Stechele, *Ber.*, 1903, **36**, 2280.

v. Braun, *Ber.*, 1902, **35**, 820.

**Tetramethylthiuram sulphide**

$C_6H_{12}N_2S_3$  MW, 208

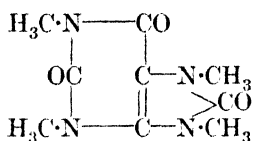
Yellow cryst. from EtOH. M.p. 104°. Very sol. EtOH, CHCl<sub>3</sub>. Spar. sol. cold Et<sub>2</sub>O. Stable to dil. acids. Alkalis → dimethylamine.

See previous references.

**Tetramethylurea.**

See under Urea.

## Tetramethyluric Acid

 $C_9H_{12}N_4O_3$ 

MW, 224

Needles from  $H_2O$ . M.p.  $228^\circ$ . Dist. without decomp. Very sol. hot  $H_2O$ , boiling  $CHCl_3$ . Mod. sol. EtOH. Spar. sol.  $Et_2O$ , cold  $H_2O$ . Decomp. by warm alkalis. Gives murexide reaction. Bitter taste.

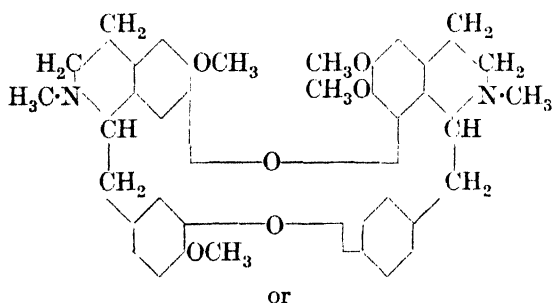
Fischer, *Ber.*, 1899, 32, 2732, 2742; 1884, 17, 1784.

## Tetraperinaphthylencyclo-octadiene.

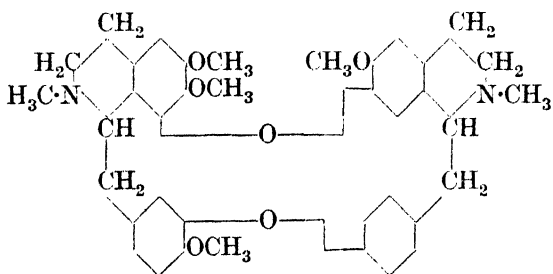
See Fluorocyclene.

## Tetrandrine

Constitution represented by



or

 $C_{38}H_{42}O_6N_2$ 

MW, 622

dl.

Colourless prisms from MeOH. M.p.  $252^\circ$ .

l.

See Phaeanthine.

d.

Alkaloid extracted from roots of *Stephania tetrandra*, S. Moore. Constituent of the Chinese drug, Han-Fang-Chi. Colourless needles. M.p.  $217-18^\circ$ . Sol.  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ . Mod. sol. EtOH. Insol.  $H_2O$ , pet. ether.  $[\alpha]_D^{26} + 252.4^\circ$  in  $CHCl_3$ . Poisonous. Affects central nervous

system, respiratory and skeletal muscles. Has action on parameria similar to that of quinine.

*B,2HCl*: prisms from EtOH. Softens at  $263^\circ$ . Decomp. at  $266^\circ$ .  $[\alpha]_D^{27} + 224.2^\circ$  in  $H_2O$ .

*B,2HBr*: needles from EtOH. Decomp. at  $270^\circ$ .  $[\alpha]_D^{27} + 200.7^\circ$  in  $H_2O$ .

*B,2HNO\_3*: prisms from EtOH.Aq. Softens at  $205^\circ$ . Decomp. at  $208^\circ$ .  $[\alpha]_D^{26} + 211.2^\circ$  in  $H_2O$ .

*B,(COOH)\_2*: needles. Softens at  $147.5-148.5^\circ$ . Decomp. at  $165-70^\circ$ .

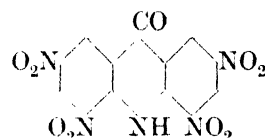
*B,2CH\_3I*: needles +  $2H_2O$  from  $Me_2CO$ .Aq. M.p.  $269^\circ$  decomp.

*Picrate*: decomp. at  $235-42^\circ$ .

Kondo, Keimatsu, *Ber.*, 1935, 68, 1505.

Chen, Chen, *J. Biol. Chem.*, 1935, 109, 681.

## 1 : 3 : 7 : 9-Tetranitroacridone

 $C_{13}H_5O_9N_3$ 

MW, 375

Yellow cryst. Does not melt below  $350^\circ$ . Sol. hot alkalis. Spar. sol. org. solvents. Sublimes. KOH  $\rightarrow$  cherry-red sol. from which K salt crystallises in red plates.

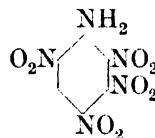
Lehmstedt, *Ber.*, 1931, 64, 2383.

Edinger, Arnold, *J. prakt. Chem.*, 1901, 64, 488.

## 2 : 4 : 5 : 7-Tetranitro-aloe-emodin.

See Aloetic Acid.

## 2 : 3 : 4 : 6-Tetranitroaniline

 $C_6H_3O_8N_5$ 

MW, 273

Yellow cryst. from AcOH. M.p.  $220^\circ$ . Slightly hygroscopic. Sol. AcOH,  $PhNO_2$ . Spar. sol.  $CHCl_3$ ,  $C_6H_6$ , ligroin. Insol. cold  $H_2O$ . Sol. 6 parts boiling  $Me_2CO$ , 3 parts *o*-nitrotoluene at  $140^\circ$ , 3 parts nitroxyline at  $150^\circ$ , and 24 parts boiling xylene. Slowly decomp. with steam at  $100^\circ$ . Warm moist  $Me_2CO$ , boiling with  $H_2O$ , or with AcONa in  $Me_2CO$ .Aq.  $\rightarrow$  2 : 4 : 6-trinitro-3-aminophenol. Boiling EtOH  $\rightarrow$  2 : 4 : 6-trinitro-3-aminophenetole. Explosive.

*N-Acetyl*: 2 : 3 : 4 : 6-tetranitroacetanilide. M.p. about  $170^\circ$  decomp.

*N-Me*: m.p.  $127^\circ$ .

*N*-Di-Me : m.p. 153°.

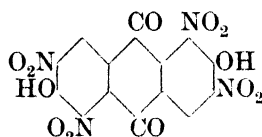
Forster, Coulson, *J. Chem. Soc.*, 1922, 121, 1992.

Duin, *Rec. trav. chim.*, 1917, 37, 115.

### Tetranitroanisole.

See under Tetranitrophenol.

**1 : 3 : 5 : 7-Tetranitroanthraflavic Acid**  
(1 : 3 : 5 : 7-Tetranitro-2 : 6-dihydroxyanthraquinone)



$C_{14}H_4O_{12}N_4$

MW, 420

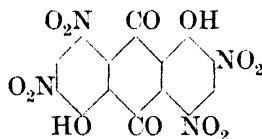
Yellow needles. Explodes at 307.6° without melting. Very sol. hot  $H_2O \rightarrow$  red sol. Sol. EtOH,  $Et_2O \rightarrow$  red sols. Gives salt with  $NH_3$ . Boiling conc.  $HNO_3 \rightarrow$  2 : 4 : 6-trinitro-*m*-hydroxybenzoic acid.

Wolffenstein, Paar, *Ber.*, 1913, 46, 596.

Schunck, Roemer, *Ber.*, 1878, 11, 1178.

Schardinger, *Ber.*, 1875, 8, 1487.

**2 : 4 : 6 : 8-Tetranitroanthrarufin** (2 : 4 : 6 : 8-Tetranitro-1 : 5-dihydroxyanthraquinone)



$C_{14}H_4O_{12}N_4$

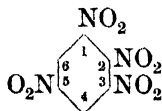
MW, 420

Yellow plates from conc.  $HNO_3$ . Gives cryst. Na, K and Mg hydrated salts. Red.  $\rightarrow$  2 : 4 : 6 : 8-tetra-aminoanthrarufin. Boiling conc.  $HNO_3 \rightarrow$  2 : 4 : 6-trinitro-*m*-hydroxybenzoic acid.

Wolffenstein, Paar, *Ber.*, 1913, 46, 592.

Liebermann, *Ber.*, 1879, 12, 188.

**1 : 2 : 3 : 5-Tetranitrobenzene**



$C_6H_2O_8N_4$

MW, 258

Yellow cryst. from  $CHCl_3$ . M.p. 126°. Mod. sol. warm  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ ,  $CS_2$ . Aq. alkalis  $\rightarrow$  picric acid.

Hollemann, *Rec. trav. chim.*, 1930, 49, 117.

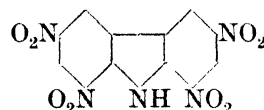
Borsche, *Ber.*, 1923, 56, 1942.

**1 : 2 : 4 : 5-Tetranitrobenzene.**

Pale yellow needles from EtOH.Aq. M.p. 188°.

Borsche, Feske, *Ber.*, 1926, 59, 820.

**1 : 3 : 6 : 8-Tetranitrocarbazole**



$C_{12}H_5O_8N_5$

MW, 347

Pale yellow plates +  $PhNO_2$  from  $PhNO_2$ , golden-yellow needles from AcOH. Darkens at 200°, m.p. 289°.

*N*-Me :  $C_{13}H_7O_8N_5$ , MW, 361. Yellow needles from  $Me_2CO$ . M.p. 277°. Insol. common org. solvents.

*N*-Et :  $C_{14}H_9O_8N_5$ , MW, 375. Cryst. from  $Me_2CO$ . M.p. 216°.

*N*-Phenyl :  $C_{18}H_9O_8N_5$ , MW, 423. Cryst. M.p. 244° (255°). Sol.  $Me_2CO$ ,  $C_6H_6$ . Insol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CCl_4$ ,  $CS_2$ .

van Alphen, *Rec. trav. chim.*, 1932, 51, 183.

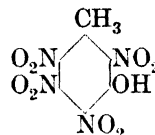
Raudnitz, *Ber.*, 1927, 60, 741.

Borsche, Scholten, *Ber.*, 1917, 50, 608.

**2 : 4 : 5 : 7-Tetranitrochrysazin.**

See Chrysammic Acid.

**2 : 4 : 5 : 6-Tetranitro-*m*-cresol**



$C_7H_4O_9N_4$

MW, 288

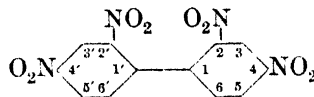
Cryst. from  $CHCl_3$ . M.p. 175°. Very sol.  $H_2O$ , EtOH  $\rightarrow$  yellow sols. Boiling  $H_2O \rightarrow$  2 : 4 : 6-trinitro-3 : 5-dihydroxytoluene. Bitter taste. Explosive.

Blanksma, *Rec. trav. chim.*, 1908, 27, 34.

**Tetranitrodihydroxyanthraquinone.**

See Tetranitroanthrarufin and Tetranitroanthraflavic Acid.

**2 : 4 : 2' : 4'-Tetranitrodiphenyl**



$C_{12}H_6O_8N_4$

MW, 334

Yellow prisms from  $C_6H_6$ . M.p. 165-6°. Very sol. AcOH,  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ .

Swann Research Inc., U.S.P., 1,870,627, (*Chem. Zentr.*, 1932, II, 2729).

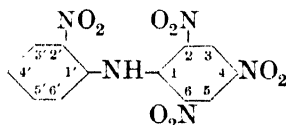
Ullmann, Bielecki, *Ber.*, 1901, 34, 2177.

**2 : 6 : 2' : 6'-Tetranitrodiphenyl.**

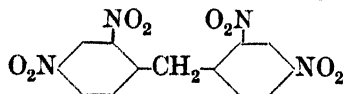
Yellowish needles from AcOH. M.p. 217-18°.

Borsche, Rantscheff, *Ann.*, 1911, **379**, 176.**3 : 4 : 2' : 4'-Tetranitrodiphenyl.**

Pale yellow cubes from MeOH. M.p. 173°.

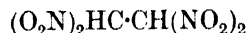
Blakey, Scarborough, *J. Chem. Soc.*, 1927, 3006.**3 : 4 : 3' : 4'-Tetranitrodiphenyl.**Yellow prisms. M.p. 186°. Very sol. AcOH, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.Ullmann, Bielecki, *Ber.*, 1901, **34**, 2179.**2 : 4 : 6 : 2'-Tetranitrodiphenylamine**C<sub>12</sub>H<sub>7</sub>O<sub>8</sub>N<sub>5</sub>

MW, 349

Yellow prismatic needles from AcOH. M.p. 234°. Sol. 600 parts toluene at 20°, 770 parts AcOH at 20°. Sol. hot dil. Na<sub>2</sub>CO<sub>3</sub> → orange-red sol.Juillard, *Bull. soc. chim.*, 1905, **33**, 1187.**2 : 4 : 6 : 3'-Tetranitrodiphenylamine.**Orange-yellow cryst. from AcOH. M.p. 213°. Spar. sol. boiling EtOH. Insol. Et<sub>2</sub>O.Duin, Lennep, *Rec. trav. chim.*, 1919, **38**, 368.Austen, *Ber.*, 1874, **7**, 1248.**2 : 4 : 6 : 4'-Tetranitrodiphenylamine.**Yellow plates or prisms from toluene. M.p. 223°. Sol. 330 parts toluene at 20°, 200 parts AcOH at 20°. Sol. hot dil. Na<sub>2</sub>CO<sub>3</sub> → red sol. Insol. cold EtOH.Duin, Lennep, *Rec. trav. chim.*, 1919, **38**, 359.**2 : 4 : 2' : 4'-Tetranitrodiphenylamine.**Reddish-brown plates from EtOH, yellow needles and prisms from AcOH. M.p. 199°. Sol. 44 parts Me<sub>2</sub>CO at 17°. Sol. NaOH → scarlet sol. Spar. sol. cold EtOH, AcOH, toluene.Ryan, Ryan, *Chem. Abstracts*, 1919, **13**, 957.Carter, *Chem. Zentr.*, 1913, II, 859.Juillard, *Bull. soc. chim.*, 1905, **33**, 1186.**2 : 4 : 2' : 4'-Tetranitrodiphenylmethane**C<sub>13</sub>H<sub>8</sub>O<sub>8</sub>N<sub>4</sub>

MW, 348

Yellow prisms from AcOH. M.p. 173°.

Matsumura, *J. Am. Chem. Soc.*, 1929, **51**, 817.**1 : 1 : 2 : 2-Tetranitroethane (sym.-Tetranitroethane)**C<sub>2</sub>H<sub>2</sub>O<sub>8</sub>N<sub>4</sub>

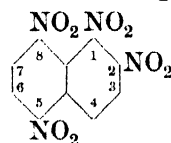
MW, 210

Di-K salt: C<sub>2</sub>O<sub>8</sub>N<sub>4</sub>K<sub>2</sub>. MW, 286. Yellow prisms with metallic reflex from MeOH.Aq. Explodes at 268° (275°) or by percussion. Spar. sol. cold H<sub>2</sub>O. Insol. MeOH, EtOH, AcOH. Aq. sol. decomp. on boiling. Cold dil. H<sub>2</sub>SO<sub>4</sub> → dinitromethane. HNO<sub>3</sub> + H<sub>2</sub>SO<sub>4</sub> → hexanitroethane.Hunter, *J. Chem. Soc.*, 1924, **125**, 1483.Will, *Ber.*, 1914, **47**, 963.**Tetranitromethane**CO<sub>8</sub>N<sub>4</sub>

MW, 196

F.p. 13° (12.5°). B.p. 125.7°, 21-3°/22 mm. (34-5°/20 mm.). Misc. with EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. D<sub>4</sub><sup>15</sup> 1.6377. n<sub>D</sub><sup>20</sup> 1.43416. Heat of comb. C. 89.6 Cal. Gives yellow col. with cyclopropane derivs. and unsaturated comps.McKie, *J. Soc. Chem. Ind.*, 1925, **44**, 430r.Berger, *Compt. rend.*, 1910, **151**, 814.Hammick, Young, *J. Chem. Soc.*, 1936, 1464.**N : 2 : 4 : 6-Tetranitro-N-methylaniline.**

See Tetryl.

**1 : 2 : 5 : 8-Tetranitronaphthalene**C<sub>10</sub>H<sub>4</sub>O<sub>8</sub>N<sub>4</sub>

MW, 308

Needles from ethyl benzoate, prisms from conc. HNO<sub>3</sub>. Decomp. at 270-310°. Spar. sol. EtOH, Me<sub>2</sub>CO, AcOH, CHCl<sub>3</sub>. Ox. → 3 : 6-dinitrophthalic acid. Red. → naphthazarin.Will, *Ber.*, 1895, **28**, 369.**1 : 2 : 6 : 8-Tetranitronaphthalene.**

White powder. Does not melt below 300°.

Dhar, *J. Chem. Soc.*, 1920, **117**, 1004.**1 : 3 : 5 : 8-Tetranitronaphthalene.**Pale yellow tetrahedra from Me<sub>2</sub>CO. M.p. 194-5°. Very sol. Me<sub>2</sub>CO, conc. HNO<sub>3</sub>. Spar. sol. EtOH, AcOH, CHCl<sub>3</sub>. Sol. alkalis → red sols. Ox. → 3 : 6-dinitrophthalic acid.

1-Naphthylamine add. comp.: black needles from  $C_6H_6$ . Decomp. at  $162^\circ$ .

2-Naphthylamine add. comp.: bronze-green needles. M.p.  $163-4^\circ$ .

Will, *Ber.*, 1895, **28**, 369.

See also previous reference.

### 1 : 3 : 6 : 8-Tetranitronaphthalene.

Yellow needles from EtOH or  $C_6H_6$ . M.p.  $207^\circ$ . Explodes on strong heating. Dil.  $HNO_3 \rightarrow$  3 : 5-dinitrophthalic acid.

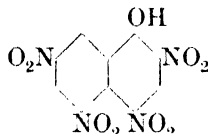
Naphthalene add. comp.: yellow needles from  $C_6H_6$ . M.p.  $191-2^\circ$ .

1-Naphthylamine add. comp.: purple needles from  $C_6H_6$ . M.p.  $204-5^\circ$ .

2-Naphthylamine add. comp.: brown plates from  $C_6H_6$ . M.p.  $211-12^\circ$ .

See previous references.

### 2 : 4 : 5 : 7-Tetranitro-1-naphthol

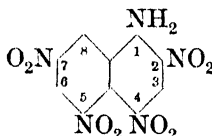


$C_{10}H_4O_9N_4$  MW, 324

Yellow plates or needles from AcOH. M.p.  $180^\circ$ . Sol. 220 parts  $C_6H_6$  at  $18^\circ$ . Spar. sol. cold AcOH. Gives cryst. Na, K, Ag, Ca and Ba salts.

Merz, Weith, *Ber.*, 1882, **15**, 2714.

### 2 : 4 : 5 : 7-Tetranitro-1-naphthylamine



$C_{10}H_5O_8N_5$  MW, 323

Pale yellow needles from EtOH or  $C_6H_6$ . M.p.  $194^\circ$ . Spar. sol. warm EtOH,  $C_6H_6$ .

N-Phenyl:  $C_{16}H_9O_8N_5$ . MW, 399. Orange-yellow needles +  $C_6H_6$  from  $C_6H_6$ , dark red needles from EtOH. M.p.  $162.5^\circ$ . Sol.  $C_6H_6$ . Spar. sol. warm EtOH,  $Et_2O$ . Boiling NaOH.Aq.  $\rightarrow$  2 : 4 : 5 : 7-tetranitro-1-naphthol.

Merz, Weith, *Ber.*, 1882, **15**, 2717.

### 2 : 4 : 5 : 8-Tetranitro-1-naphthylamine.

Yellow needles from EtOH. M.p.  $202^\circ$ .

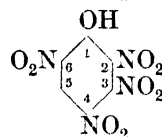
N-Phenyl: orange needles +  $C_6H_6$  from  $C_6H_6$ , dark red needles from EtOH. M.p.  $253^\circ$ . Spar. sol. warm EtOH,  $C_6H_6$ .

See previous reference.

### Tetranitrophenetole.

See under Tetranitrophenol.

### 2 : 3 : 4 : 6-Tetranitrophenol



$C_6H_2O_9N_4$

MW, 274

Pale yellow cryst. from  $CHCl_3$ . M.p.  $140^\circ$ .

Me ether: 2 : 3 : 4 : 6-tetranitroanisole.  $C_7H_4O_8N_4$ . MW, 288. Leaflets. M.p.  $94^\circ$ . Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ .

Blanksma, *Rec. trav. chim.*, 1902, **21**, 256.

Claessen, D.R.P., 289,446, (*Chem. Zentr.*, 1916, I, 240).

### 2 : 3 : 5 : 6-Tetranitrophenol.

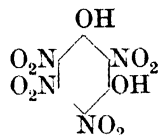
Me ether: 2 : 3 : 5 : 6-tetranitroanisole. Exists in two forms. (i) Cryst. from EtOH. M.p.  $154^\circ$ . (ii) Pale yellow needles from  $C_6H_6$ . M.p.  $112^\circ$ .

Et ether: 2 : 3 : 5 : 6-tetranitrophenetole.  $C_8H_6O_9N_4$ . MW, 302. Yellow cryst. from EtOH. M.p.  $115^\circ$ .

Blanksma, *Rec. trav. chim.*, 1904, **23**, 114; 1905, **24**, 42.

Devergnes, *Chem. Abstracts*, 1929, **23**, 4207.

### Tetranitroresorcinol



$C_6H_2O_{10}N_4$

MW, 290

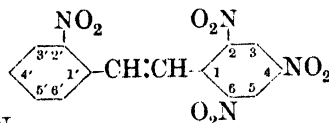
Cryst. from  $CHCl_3$  or  $CCl_4$ . M.p.  $152^\circ$ . Bitter taste. Explosive.

Mono-Me ether:  $C_7H_4O_{10}N_4$ . MW, 304. Cryst. from  $CHCl_3$  or  $CCl_4$ . M.p.  $115^\circ$ . Bitter taste. Explosive.

Mono-Et ether:  $C_8H_6O_{10}N_4$ . MW, 318. Cryst. from  $CHCl_3$  or  $CCl_4$ . M.p.  $110^\circ$ . Bitter taste. Explosive.

Blanksma, *Rec. trav. chim.*, 1908, **27**, 35.

### 2 : 4 : 6 : 2'-Tetranitrostilbene



$C_{14}H_8O_8N_4$

MW, 360

Pale brown needles from EtOH. M.p.  $181^\circ$ .

Bishop, Brady, *J. Chem. Soc.*, 1922, 2367.



**2 : 4 : 6 : 3'-Tetranitrostilbene.**

Lemon-yellow leaflets from  $\text{Me}_2\text{CO}-\text{C}_6\text{H}_6$ .  
M.p. 159°.

See previous reference.

**2 : 4 : 6 : 4'-Tetranitrostilbene.**

Yellow needles. M.p. 196°. Sol. AcOH. Spar. sol. EtOH,  $\text{C}_6\text{H}_6$ .

Ullmann, Gschwind, *Ber.*, 1908, **41**, 2297.

**2 : 4 : 2' : 4'-Tetranitrostilbene.**

Yellow needles from AcOH or  $\text{PhNO}_2$ . M.p. 266-7° decomp. Insol. Et<sub>2</sub>O.

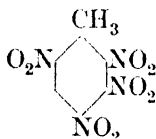
Escalas, *Ber.*, 1904, **37**, 3599.

Green, Baddiley, *J. Chem. Soc.*, 1908, **93**, 1725.

**2 : 6 : 2' : 6'-Tetranitrostilbene.**

Yellow needles from  $\text{PhNO}_2$ . M.p. 250°. Sol.  $\text{Me}_2\text{CO}$ . Insol. EtOH, Et<sub>2</sub>O, ligroin.

Reich, Wetter, Widmer, *Ber.*, 1912, **45**, 3059.

**2 : 3 : 4 : 6-Tetranitrotoluene**

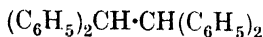
$\text{C}_7\text{H}_4\text{O}_8\text{N}_4$  MW, 272

Cryst. from  $\text{CHCl}_3$  or conc.  $\text{HNO}_3$ . M.p. 136-5°.

Holleman, *Rec. trav. chim.*, 1930, **49**, 501.

**Tetraphenyldiarsine.**

See Phenylacetyl.

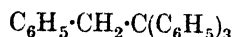
**sym.-Tetraphenylethane**

$\text{C}_{26}\text{H}_{22}$  MW, 334

Prisms from  $\text{CHCl}_3$ . M.p. 211°. B.p. 358-62°, 260°/16 mm.

Montagne, *Rec. trav. chim.*, 1906, **25**, 407.

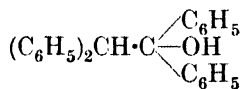
Morris, Thomas, Brown, *Ber.*, 1910, **43**, 2959.

**unsym.-Tetraphenylethane** ( $\alpha$ -Benzyl-tritan, triphenylbenzylmethane)

$\text{C}_{26}\text{H}_{22}$  MW, 334

Plates from AcOEt. M.p. 144°. B.p. 277-80°/21 mm. Mod. sol.  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, Et<sub>2</sub>O.

Gomberg, Cone, *Ber.*, 1906, **39**, 1463.

**1 : 1 : 2 : 2 - Tetraphenylethyl Alcohol**  
(Hydroxy-sym.-tetraphenylethane,  $\alpha$ -benzhydryl-benzhydrol)

$\text{C}_{26}\text{H}_{22}\text{O}$  MW, 350

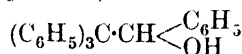
Needles from  $\text{C}_6\text{H}_6$ . M.p. 326°. Mod. sol. EtOH, Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ , ligroin.

*Benzoyl*: prisms from AcOEt. M.p. 155°.

*Phenylurethane*: cryst. from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 163-5°.

Richard, *Compt. rend.*, 1934, **198**, 1242.

Bergmann, Wagenberg, *Ber.*, 1930, **63**, 2591.

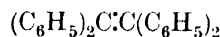
**1 : 2 : 2 : 2 - Tetraphenylethyl Alcohol**  
( $\omega$ -Triphenylmethylbenzyl alcohol, hydroxy-unsym.-tetraphenylethane)

$\text{C}_{26}\text{H}_{22}\text{O}$  MW, 350

Plates from EtOH. M.p. 151°.

*Acetyl*: cryst. from EtOH. M.p. 131°.

Schlenk, Ochs, *Ber.*, 1916, **49**, 611.

**Tetraphenylethylene**

$\text{C}_{26}\text{H}_{20}$  MW, 332

Plates from EtOH- $\text{C}_6\text{H}_6$ . M.p. 223-4° (220-1°). B.p. 415-25°. Sol.  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH, Et<sub>2</sub>O.

Mackenzie, *J. Chem. Soc.*, 1922, **121**, 1697.

**Tetraphenylethylene Glycol.**

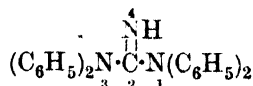
See Benzpinacol.

**Tetraphenylethylene oxide.**

See Benzpinacolin.

**Tetraphenylfuran.**

See Lepidene.

**1 : 1 : 3 : 3-Tetraphenylguanidine**

$\text{C}_{25}\text{H}_{21}\text{N}_3$  MW, 363

Cryst. from ligroin. M.p. 130-1°. Sol. EtOH, Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ .

4-N-Benzoyl: cryst. from EtOH. M.p. 142-4°.

Weith, *Ber.*, 1874, **7**, 843.

Johnson, Chemoff, *J. Am. Chem. Soc.*, 1912, **34**, 170.

**1 : 1 : 3 : 4-Tetraphenylguanidine**

737

**1 : 2 : 3 : 3-Tetraphenylpropyl Alcohol****1 : 1 : 3 : 4-Tetraphenylguanidine**

$$\text{C}_{25}\text{H}_{21}\text{N}_3 \quad \text{C}_6\text{H}_5 \cdot \text{NH} \cdot \overset{\text{N} \cdot \text{C}_6\text{H}_5}{\underset{|}{\text{C}}} \cdot \text{N}(\text{C}_6\text{H}_5)_2 \quad \text{MW, 363}$$
 Cryst. from EtOH. M.p. 137–40°. *Chloroplatinate* : m.p. 240–2°. Steindorff, *Ber.*, 1904, **37**, 964.

**Tetraphenylhydrazine**

$$\text{C}_{24}\text{H}_{20}\text{N}_2 \quad (\text{C}_6\text{H}_5)_2\text{N} \cdot \text{N}(\text{C}_6\text{H}_5)_2 \quad \text{MW, 336}$$
 Prisms from  $\text{CHCl}_3$ –EtOH. M.p. 147° decomp. Sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. EtOH.

Chattaway, Ingle, *J. Chem. Soc.*, 1895, **67**, 1091.

Wieland, Gambarjan, *Ber.*, 1906, **39**, 1500.

**1 : 1 : 2 : 3-Tetraphenylisopropyl Alcohol**  
(2-Hydroxy-1 : 1 : 2 : 3-tetraphenylpropane)

$$\text{C}_{27}\text{H}_{24}\text{O} \quad \text{C}_6\text{H}_5 \cdot \text{CH}_2 \cdot \overset{\text{C}_6\text{H}_5}{\underset{|}{\text{C}}}(\text{OH}) \cdot \text{CH}(\text{C}_6\text{H}_5)_2 \quad \text{MW, 364}$$
 Needles from ligroin. M.p. 135–6°. Sol. EtOH,  $\text{C}_6\text{H}_6$ . Mod. sol. ligroin. *Acetyl* : needles from benzene. M.p. 151–3°. Bergmann, Weiss, *Ber.*, 1931, **64**, 1489. Orékhoff, *Bull. soc. chim.*, 1919, **25**, 186.

**Tetraphenylmethane**

$$\text{C}_{25}\text{H}_{20} \quad \text{C}(\text{C}_6\text{H}_5)_4 \quad \text{MW, 320}$$
 Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 282°. B.p. 431°. Insol.  $\text{Et}_2\text{O}$ , AcOH, ligroin. Sublimes in needles. Sol.  $\text{H}_2\text{SO}_4$  with red col.

Gomberg, Kamm, *J. Am. Chem. Soc.*, 1917, **39**, 2009.

Gomberg, Cone, *Ber.*, 1906, **39**, 1463.

**Tetraphenylphenylenediamine.**

See under Phenylenediamine.

**Tetraphenylphosphonium halides.**

See under Triphenylphosphine.

**1 : 1 : 1 : 3-Tetraphenylpropane**

$$\text{C}_{27}\text{H}_{24} \quad \text{C}_6\text{H}_5 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C}(\text{C}_6\text{H}_5)_3 \quad \text{MW, 348}$$
 Prisms from EtOH. M.p. 126°.

Wieland, Kloss, *Ann.*, 1929, **470**, 214.

Dict. of Org. Comp.—III.

**1 : 1 : 2 : 3-Tetraphenylpropane**

$$\text{C}_{27}\text{H}_{24} \quad \text{C}_6\text{H}_5 \cdot \text{CH}_2 \cdot \overset{\text{C}_6\text{H}_5}{\underset{|}{\text{C}}} \cdot \text{CH}(\text{C}_6\text{H}_5)_2 \quad \text{MW, 348}$$
 Needles from EtOH. M.p. 87–9°.

Bergmann, Weiss, *Ber.*, 1931, **64**, 1491.

**1 : 1 : 3 : 3-Tetraphenylpropane**

$$\text{C}_{27}\text{H}_{24} \quad (\text{C}_6\text{H}_5)_2\text{CH} \cdot \text{CH}_2 \cdot \text{CH}(\text{C}_6\text{H}_5)_2 \quad \text{MW, 348}$$
 Needles from EtOH. M.p. 139°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

Wittig, Obermann, *Ber.*, 1934, **67**, 2056.

Vorländer, Siebert, *Ber.*, 1906, **39**, 1028.

**1 : 1 : 2 : 3-Tetraphenylpropyl Alcohol**  
(1-Hydroxy-1 : 1 : 2 : 3-tetraphenylpropane)

$$\text{C}_{27}\text{H}_{24}\text{O} \quad \text{C}_6\text{H}_5 \cdot \text{CH}_2 \cdot \overset{\text{C}_6\text{H}_5}{\underset{|}{\text{C}}}(\text{OH}) \cdot \text{CH}(\text{C}_6\text{H}_5)_2 \quad \text{MW, 364}$$
 Needles from EtOH. M.p. 165°.

Sernagiotto, *Chem. Abstracts*, 1920, **14**, 1672.

**1 : 1 : 3 : 3-Tetraphenylpropyl Alcohol**  
(1-Hydroxy-1 : 1 : 3 : 3-tetraphenylpropane)

$$\text{C}_{27}\text{H}_{24}\text{O} \quad (\text{C}_6\text{H}_5)_2\text{CH} \cdot \text{CH}_2 \cdot \text{C}(\text{OH})(\text{C}_6\text{H}_5)_2 \quad \text{MW, 364}$$
 Needles. M.p. 95–6°. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. ligroin.

Kohler, *Ann. Chem. J.*, 1904, **31**, 651.

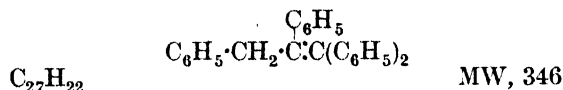
**1 : 2 : 2 : 3-Tetraphenylpropyl Alcohol**  
(1-Hydroxy-1 : 2 : 2 : 3-tetraphenylpropane)

$$\text{C}_{27}\text{H}_{24}\text{O} \quad \text{C}_6\text{H}_5 \cdot \text{CH}_2 \cdot \overset{\text{C}_6\text{H}_5}{\underset{\text{C}_6\text{H}_5}{\underset{|}{\text{C}}}} \cdot \text{CH}(\text{OH}) \cdot \text{C}_6\text{H}_5 \quad \text{MW, 364}$$
 Cryst. from pet. ether. M.p. 141–2°. Schlenk *et al.*, *Ann.*, 1928, **463**, 261.

**1 : 2 : 3 : 3-Tetraphenylpropyl Alcohol**  
(1-Hydroxy-1 : 2 : 3 : 3-tetraphenylpropane)

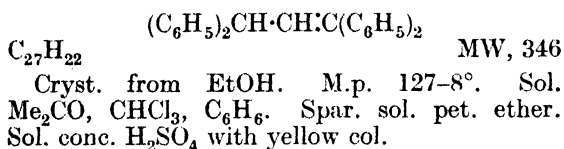
$$\text{C}_{27}\text{H}_{24}\text{O} \quad (\text{C}_6\text{H}_5)_2\text{CH} \cdot \overset{\text{C}_6\text{H}_5}{\underset{|}{\text{C}}} \cdot \text{CH}(\text{OH}) \cdot \text{C}_6\text{H}_5 \quad \text{MW, 364}$$
 Cryst. from  $\text{Et}_2\text{O}$ . M.p. 132°. Sol. EtOH, AcOH. Mod. sol.  $\text{Et}_2\text{O}$ .

Japp, Klingemann, *J. Chem. Soc.*, 1890, **57**, 669.

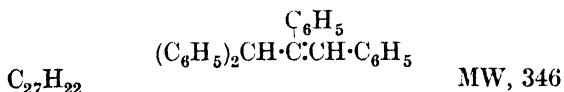
**1 : 1 : 2 : 3-Tetraphenylpropylene** (1 : 1 : 2-Triphenyl-2-benzylethylene)

Needles from methyl ethyl ketone. M.p. 142°. Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CCl}_4$ . Spar. sol.  $\text{Et}_2\text{O}$ . Prac. insol. cold  $\text{EtOH}$ ,  $\text{AcOH}$ .

Bergmann, Weiss, *Ber.*, 1931, **64**, 1489.  
Meisenheimer, Schlichenmaier, *Ann.*, 1927, **456**, 151.

**1 : 1 : 3 : 3-Tetraphenylpropylene**

Wittig, Obermann, *Ber.*, 1934, **67**, 2054.  
Vorländer, Siebert, *Ber.*, 1906, **39**, 1032.

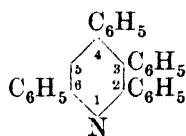
**1 : 2 : 3 : 3-Tetraphenylpropylene**

Needles from  $\text{EtOH}$ . M.p. 131°.

Bergmann, Weiss, *Ber.*, 1931, **64**, 1489.

**Tetraphenylpyrazine.**

See Amaron.

**2 : 3 : 4 : 6-Tetraphenylpyridine**

Needles from  $\text{EtOH}$ . M.p. 182°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ . Insol. ligroin.

Picrate: yellow needles from  $\text{Et}_2\text{O}$ . M.p. 192°.

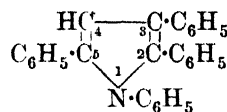
Dilthey, Böttler, *Ber.*, 1919, **52**, 2048.

Dilthey, Nüsslein, Meyer, Kaffer, *J. prakt. Chem.*, 1922, **104**, 33.

**2 : 3 : 5 : 6-Tetraphenylpyridine.**

Prisms from  $\text{EtOH}-\text{C}_6\text{H}_6$ . M.p. 233.5°. Sol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ . Mod. sol.  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{AcOH}$ , ligroin. Sol. conc.  $\text{H}_2\text{SO}_4$  with blue fluor.

Carpenter, *Ann.*, 1898, **302**, 233.

**1 : 2 : 3 : 5-Tetraphenylpyrrole**

Needles from  $\text{AcOH}$ . M.p. 197°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{EtOH}$ ,  $\text{AcOH}$ .

Smith, *J. Chem. Soc.*, 1890, **57**, 646.

**2 : 3 : 4 : 5-Tetraphenylpyrrole.**

Needles from  $\text{AcOH}$ . M.p. 214–15°. Prac. insol.  $\text{EtOH}$ . Sol. conc.  $\text{H}_2\text{SO}_4$  with yellow col.

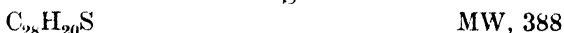
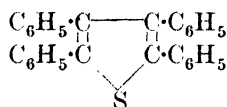
*N-Me*:  $\text{C}_{29}\text{H}_{23}\text{N}$ . MW, 385. Plates from  $\text{Et}_2\text{O}$ . M.p. 214°. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

*N-Et*:  $\text{C}_{30}\text{H}_{25}\text{N}$ . MW, 399. Needles from  $\text{CHCl}_3$ . M.p. 221°.

*N-Acetyl*: needles from  $\text{AcOH}$  or  $\text{C}_6\text{H}_6$ . M.p. 226°. Prac. insol.  $\text{EtOH}$ .

Robinson, Robinson, *J. Chem. Soc.*, 1918, **113**, 644.

Fehrlin, *Ber.*, 1889, **22**, 555.

**Tetraphenylthiophene (Thionessal)**

Needles from  $\text{EtOH}$ . M.p. 184–5°. B.p. about 400°. Mod. sol.  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin. Very spar. sol.  $\text{EtOH}$ .

Bergman, *J. Chem. Soc.*, 1936, 505.

Dilthey *et al.*, *Ber.*, 1935, **68**, 1159.

**Tetraphenylthiourea.**

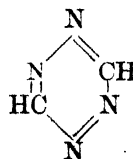
See under Thiourea.

**Tetraphenylurea.**

See under Urea.

**Tetraphthene.**

See Tetrahydroacenaphthene

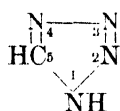
**1 : 2 : 4 : 5-Tetrazine**

Crimson cryst. M.p. 99°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, liq. NH<sub>3</sub>. Sol. H<sub>2</sub>SO<sub>4</sub> with red col. Sublimes. Volatile at room temp.

Wood, Bergstrom, *J. Am. Chem. Soc.*, 1933, **55**, 3649.

Curtius, Darapsky, Müller, *Ber.*, 1907, **40**, 84.

### 1 : 2 : 3 : 4-Tetrazole (*Pyrro- $\alpha\beta\beta'$ -triazole*)



CH<sub>2</sub>N<sub>4</sub> MW, 70

Plates from EtOH. M.p. 156°. Sol. H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO, AcOH. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Sublimes. Salts are explosive.

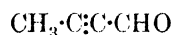
*N-Me*: see 1-Methyl-1 : 2 : 3 : 4-tetrazole.

*N-Phenyl*: see 1-Phenyl-1 : 2 : 3 : 4-tetrazole.

Freund, Paradies, *Ber.*, 1901, **34**, 3110.

Pechmann, Wedekind, *Ber.*, 1895, **28**, 1693.

**Tetrolaldehyde** (*Allylene-1-aldehyde, methylacetylene-aldehyde*)



C<sub>4</sub>H<sub>4</sub>O MW, 68

F.p. — 26°. B.p. 106.5–107°, 27–8°/34 mm. D<sub>4</sub><sup>0</sup> 0.944, D<sub>4</sub><sup>17</sup> 0.9265. n<sub>D</sub><sup>17</sup> 1.4467. Readily oxidises in air.

*Oxime*: needles. M.p. 108–9°.

*Semicarbazone*: m.p. 158°.

*Hydrazone*: liq. B.p. 170° decomp., 63–5°/15 mm. D<sub>4</sub><sup>18.5</sup> 0.9768. n<sub>D</sub><sup>18.5</sup> 1.530. Misc. with H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

*p-Nitrophenylhydrazone*: yellow needles from EtOH.Aq. M.p. 157–8°.

*Azine*: yellow needles from EtOH. M.p. 123–4°.

*Di-Me acetal*: b.p. 144.5°. D<sub>15</sub> 0.954.

*Di-Me acetal*: b.p. 169–70°, 62–5°/15 mm. D<sub>16</sub> 0.9012. n<sub>D</sub><sup>16</sup> 1.4269.

Claisen, *Ber.*, 1911, **44**, 1166.

Viguiet, *Compt. rend.*, 1911, **152**, 1491.

**Tetrollic Acid** (*Methylacetylene-carboxylic acid, methylpropionic acid, allylene-1-carboxylic acid*)



C<sub>3</sub>H<sub>4</sub>O<sub>2</sub> MW, 72

Plates from Et<sub>2</sub>O or CS<sub>2</sub>. M.p. 77–8°. B.p. 203°, 99–100°/18 mm.  $k = 2.46 \times 10^{-3}$  at 25°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>. Volatile in steam. At 211° → allylene.

*Et ester*: C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>. MW, 100. Oil. B.p. 163–4°. D<sub>4</sub><sup>24.1</sup> 0.9621. n<sub>D</sub><sup>24.1</sup> 1.43495.

*Chloride*: C<sub>3</sub>H<sub>3</sub>OCl. MW, 90.5. Liq. Fumes in air. Very unstable.

*Amide*: C<sub>3</sub>H<sub>5</sub>ON. MW, 71. Cryst. from EtOH. M.p. 147–8°. Mod. sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O. Volatile in steam.

Feist, *Ann.*, 1906, **345**, 104.

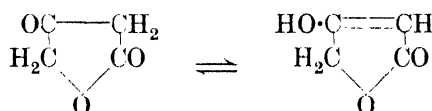
Auwers, *Ber.*, 1935, **68**, 1637.

Bourguel, Yvon, *Bull. soc. chim.*, 1929, **45**, 1067.

### Tetronal.

See Ethylsulphonal.

**Tetronic Acid** (*Dihydroxycrotonic lactone, 3-hydroxyacetoacetic lactone, 2-ketobutyrolactone, 2 : 4-diketotetrahydrofuran*)



C<sub>4</sub>H<sub>4</sub>O<sub>3</sub> MW, 100

Plates from EtOH-ligroin. Sinters at 135°. M.p. 141°. Very sol. H<sub>2</sub>O, warm EtOH. Spar. sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, ligroin, C<sub>6</sub>H<sub>6</sub>. Strong monobasic acid. Decomposes bicarbonates. FeCl<sub>3</sub> → dark red col. Heat conc. aq. sol. → anhydrotetronic acid.

*Anhydride*: anhydrotetronic acid. C<sub>8</sub>H<sub>6</sub>O<sub>2</sub>. MW, 182. Needles + 1H<sub>2</sub>O from hot H<sub>2</sub>O. M.p. 263° decomp. Sol. EtOH. Spar. sol. cold H<sub>2</sub>O, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Decomposes bicarbonates. FeCl<sub>3</sub> → red. col.

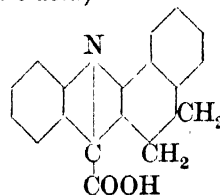
*Oxime*: plates from hot EtOH. Decomp. at 146°. Very sol. H<sub>2</sub>O. Sol. EtOH, Et<sub>2</sub>O. No col. with FeCl<sub>3</sub>.

*Benzoyl*: plates or prisms from CHCl<sub>3</sub>-ligroin. M.p. 120°.

*Phenylhydrazone*: prisms from EtOH.Aq. M.p. 128°.

Wolff, Schwabe, *Ann.*, 1896, **291**, 234.

**Tetrophane** (3 : 4-Dihydro-1 : 2-naphthacridine-14-carboxylic acid)



C<sub>18</sub>H<sub>13</sub>O<sub>2</sub>N MW, 275

Yellow needles. M.p. 252° decomp. to 3 : 4-dihydro-1 : 2-naphthacridine. Very sol. tetralin.

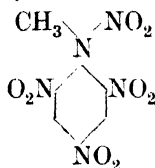
Mod. sol. AcOH. Spar. sol. EtOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*Et ester*: C<sub>20</sub>H<sub>17</sub>O<sub>2</sub>N. MW, 303. Needles. M.p. 80°.

*Amide*: C<sub>18</sub>H<sub>14</sub>ON<sub>2</sub>. MW, 274. Colourless powder from EtOH-pet. ether. M.p. 220-2°.

v. Braun, Wolff, *Ber.*, 1922, 55, 3679.

**Tetryl** (N-Nitro-N-methyl-2 : 4 : 6-trinitro-aniline, methylpicrylnitramine)



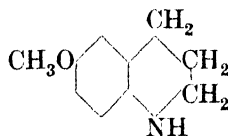
C<sub>7</sub>H<sub>5</sub>O<sub>8</sub>N<sub>5</sub> MW, 287

Yellow prisms from EtOH. M.p. 131-2°. D<sub>19</sub> 1.57. Sol. Me<sub>2</sub>CO, Py. Mod. sol. AcOEt, C<sub>6</sub>H<sub>6</sub>. Spar. sol. MeOH, EtOH. Explodes on heating to about 180-90°. NaOH → picric acid. Used as explosive chiefly in admixture with other explosives such as trinitrotoluene (T.N.T.).

van Duin, *Rec. trav. chim.*, 1918, 37, 112.

Tanner, *Chem. Met. Eng.*, 1923, 29, 404.

**Thalline** (6-Methoxy-1 : 2 : 3 : 4-tetrahydroquinoline)



C<sub>10</sub>H<sub>13</sub>ON MW, 163

Rhombic cryst. from H<sub>2</sub>O, prisms from EtOH or pet. ether, m.p. 42-3°. B.p. 283°/735 mm. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O, pet. ether. Difficultly volatile in steam. HCl, H<sub>2</sub>SO<sub>4</sub> and AcOH salts have antipyretic action. Ox. agents → emerald green col. FeCl<sub>3</sub> on boiling → brownish-green or rose col.

*B<sub>2</sub>HI*: prisms from EtOH. M.p. 155-6°.

*N-Me*: C<sub>11</sub>H<sub>15</sub>ON. MW, 177. Oil. B.p. 227-228.5°, 150-1°/10 mm. Dil. HCl sol. + FeCl<sub>3</sub> → dark red col. Cl water → reddish-yellow col. *Methiodide*: needles + 1H<sub>2</sub>O from H<sub>2</sub>O, prisms from EtOH. M.p. 223-4° decomp. *Picrate*: yellow plates. M.p. 164°.

*N-Et*: C<sub>12</sub>H<sub>17</sub>ON. MW, 191. Yellow oil. B.p. 287-287.5° slight decomp. Misc. with EtOH, Et<sub>2</sub>O. Very sol. min. acids. Insol. H<sub>2</sub>O. *Ethiodide*: needles. M.p. 131-3°.

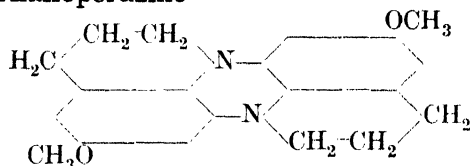
*N-Allyl*: C<sub>13</sub>H<sub>17</sub>ON. MW, 203. Yellow oil. B.p. 176°/12 mm.

*N-Acetyl*: prisms from Et<sub>2</sub>O-pet. ether. M.p. 46-7°.

*Picrate*: yellow needles. M.p. 162°.

Skraup, *Monatsh.*, 1885, 6, 767.

### Thallopiperazine

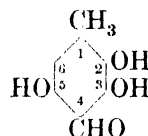


C<sub>20</sub>H<sub>22</sub>O<sub>2</sub>N<sub>2</sub> MW, 322

Yellow cryst. from AcOEt. M.p. 160°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. Et<sub>2</sub>O. Spar. sol. AcOEt. cold EtOH. H<sub>2</sub>SO<sub>4</sub> → red sol. with reddish-green fluorescence; with H<sub>2</sub>O, violet → pink.

Wieland, Haas, *Ber.*, 1920, 53, 1342.

**Thamnol** (2 : 3 : 5-Trihydroxy-p-toluic aldehyde)



C<sub>8</sub>H<sub>8</sub>O<sub>4</sub> MW, 168

Yellow prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 186°. Sol. EtOH, Et<sub>2</sub>O, AcOEt, Py. Spar. sol. cold C<sub>6</sub>H<sub>6</sub>. Sol. H<sub>2</sub>O → acid sol. Sol. alkalis → intense yellow col. Alc. FeCl<sub>3</sub> → green col.

*3 : 5-Di-Me ether*: C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>. MW, 196. Yellow prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 104°.

*Triacetyl*: needles from EtOH. M.p. 133°.

*Anil*: brownish leaflets. M.p. 128-9°.

*Phenylhydrazone*: brownish-red spears from EtOH. M.p. 194° decomp.

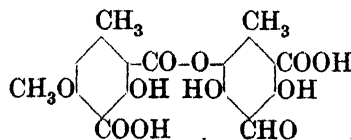
*p-Nitrophenylhydrazone*: brownish-violet needles from EtOH.Aq. M.p. about 320° decomp.

Koller, Hamburg, *Monatsh.*, 1935, 65, 378.

Asahina, Fuzikawa, *Ber.*, 1932, 65, 58.

Asahina, Ihara, *Ber.*, 1929, 62, 1204.

### Thamnolic Acid



C<sub>19</sub>H<sub>16</sub>O<sub>11</sub> MW, 420

Constituent of many lichens. Colourless prisms from Me<sub>2</sub>CO. M.p. 223°. Sol. alkalis and alk. carbonates → yellow sols. Alc. FeCl<sub>3</sub> →

brownish-red col. Loses  $\text{CO}_2$  on heating to  $120\text{--}30^\circ$  with  $\text{Me}_2\text{CO}$ .

*Anil.*: orange-yellow cryst. from EtOH. M.p.  $206\text{--}8^\circ$ .

*Phenyldiazone*: yellow prisms from EtOH. M.p.  $173\text{--}4^\circ$ .

*p*-Nitrophenylhydrazone: orange-yellow cryst. from EtOH. M.p.  $238\text{--}9^\circ$ .

Asahina, Hiraiwa, *Ber.*, 1936, **69**, 330.

Asahina, Ihara, *Ber.*, 1929, **62**, 1196.

**Thapsic Acid** (*Thapsiaic acid*, 1:14-tetra-decane-dicarboxylic acid)



$\text{C}_{16}\text{H}_{30}\text{O}_4$

MW, 286

Occurs as ester in roots of *Thapsia garganica*, Linn., and in wax of *Juniperus sabina*. Plates from EtOH or AcOEt. M.p.  $126^\circ$ . Sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Distils undecomp.  $\text{Ac}_2\text{O} \rightarrow$  anhydride.

*Di-Me ester*:  $\text{C}_{18}\text{H}_{34}\text{O}_4$ . MW, 314. M.p.  $51\text{--}2^\circ$ . B.p.  $150\text{--}60^\circ/0.3$  mm.

*Di-Et ester*:  $\text{C}_{20}\text{H}_{38}\text{O}_4$ . MW, 342. Cryst. M.p.  $39^\circ$ . B.p.  $160\text{--}5^\circ/0.3$  mm. Very sol.  $\text{Et}_2\text{O}$ , EtOH. Spar. sol. pet. ether.

*Anhydride*:  $\text{C}_{16}\text{H}_{28}\text{O}_3$ . MW, 268. Cryst. powder from  $\text{C}_6\text{H}_6$ . M.p.  $71^\circ$ . Boiling  $\text{H}_2\text{O} \rightarrow$  thapsic acid.

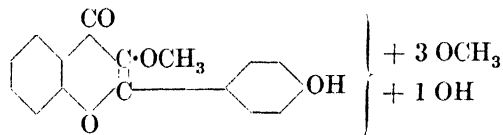
*Dianilide*: cryst. from MeOH. M.p.  $163^\circ$ .

Schmid, Kemeny, *Monatsh.*, 1935, **66**, 3.

Bougault, *Compt. rend.*, 1910, **150**, 875.

Canzoneri, *Gazz. chim. ital.*, 1883, **13**, 516.

### Thapsin (*Calycopterin*)



$\text{C}_{19}\text{H}_{18}\text{O}_8$

MW, 374

Occurs in Spanish fox-glove (*Digitalis thapsi*, Linn.), and leaves of *Calycopteris floribunda*, Lamk. Yellow prisms from AcOH, needles from  $\text{Et}_2\text{O}$ . M.p.  $225\text{--}6^\circ$ . Very sol.  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ . Sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ , pet. ether. Alkalis  $\rightarrow$  yellow sols.  $\text{FeCl}_3 \rightarrow$  green col.  $\text{HI} \rightarrow$  calycopteretin.  $\text{KOH}$  fusion  $\rightarrow$  *p*-hydroxybenzoic acid.

*Di-Me ether*:  $\text{C}_{21}\text{H}_{22}\text{O}_8$ . MW, 402. Yellow prisms or rhombohedra from EtOH. M.p.  $130^\circ$ .

*Di-Et ether*:  $\text{C}_{23}\text{H}_{26}\text{O}_8$ . MW, 430. Yellow cryst. from EtOH. M.p.  $130^\circ$ .

*Diacetyl*: colourless plates or needles from EtOH. M.p.  $129^\circ$ .

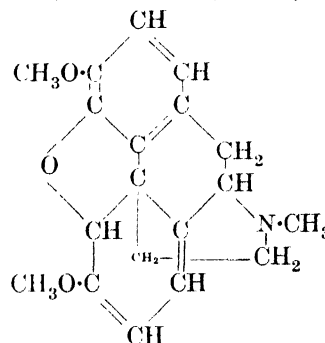
*Dibenzoyl*: m.p.  $165^\circ$ .

Karrer, Venkataraman, *Nature*, 1935, **135**, 878.

Ratnagiriswaran, Sehra, Venkataraman, *Biochem. J.*, 1934, **28**, 1964.

Karrer, *Helv. Chim. Acta*, 1934, **17**, 1560.

**Thebaine** (*Methyl ether of enol of codeinone*)



$\text{C}_{19}\text{H}_{21}\text{O}_3\text{N}$

MW, 311

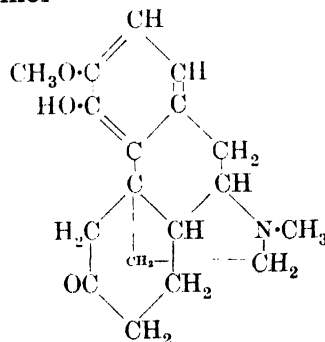
Plates from EtOH.Aq., prisms from EtOH. M.p.  $193^\circ$ . Very sol. EtOH,  $\text{CHCl}_3$ . Sol. 140 parts  $\text{Et}_2\text{O}$  at  $10^\circ$ , 18 parts cold  $\text{C}_6\text{H}_6$ . Sol.  $\text{H}_2\text{SO}_4 \rightarrow$  red col. Tasteless.  $[\alpha]_D^{25} = 218.6^\circ$  in EtOH. Decomp. by dil. min. acids. Dil.  $\text{HCl} \rightarrow$  thebenin. Very poisonous.

*Picrate*: m.p.  $217^\circ$ .

v. Braun, Cahn, *Ann.*, 1926, **451**, 55.

Schöpf, Winterholder, *Ann.*, 1927, **452**, 232.

### Thebainol



$\text{C}_{18}\text{H}_{23}\text{O}_3\text{N}$

MW, 301

Cryst. + MeOH from MeOH, m.p.  $50\text{--}4^\circ$ ; colourless prisms from  $\text{Et}_2\text{O}$ , m.p.  $135\text{--}6^\circ$ .  $[\alpha]_D^{25} + 67.05^\circ$  in EtOH. Sol. dil. alkalis. Forms cryst. Na salt. Forms benzylidene and piperonylidene derivs.

*Oxime*: cryst. from EtOH. M.p.  $217\text{--}18^\circ$ .  $[\alpha]_D^{25} + 104.2^\circ$  in AcOH.Aq.

*Semicarbazone*: needles from EtOH-AcOEt. M.p. 217-18°.

*Perchlorate*: cryst. from EtOH. M.p. 245°.

*Methiodide*: prisms from EtOH. M.p. 243° decomp.

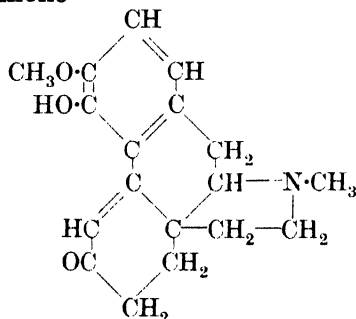
Gulland, *J. Chem. Soc.*, 1928, 706.

Schöpf, Borkowsky, *Ann.*, 1927, 458, 170.

Gulland, Robinson, *J. Chem. Soc.*, 1923, 123, 998.

Pschorr, *Ber.*, 1905, 38, 3167.

### Thebainone



$C_{18}H_{21}O_3N$

MW, 299

Colourless needles +  $\frac{1}{2}H_2O$  from EtOH.Aq. M.p. 151-2°. Loses  $H_2O$  at 100° in vacuo. Very sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ . Sol. 250 parts cold  $H_2O$ , 120 parts hot. Sol. 4 parts EtOH, 5 parts AcOEt. Yellow aq. sol. reacts alkaline. Sol. alkalis  $\rightarrow$  yellow col. Sol. conc. HCl,  $H_2SO_4$ . Forms benzyldine and piperonylidene derivs.

*B,HI*: needles from  $H_2O$ . M.p. 258-9°.

*Me ether*:  $C_{19}H_{23}O_3N$ . MW, 313. Prisms from MeOH. M.p. 156°. *Methiodide*: plates from EtOH. M.p. 256°.

*Oxime*: prisms +  $\frac{1}{2}H_2O$  from EtOH.Aq. M.p. 200-1° (185-6°). *B,HCl*: needles from  $H_2O$ . M.p. 290-1°.

*Semicarbazone*: needles from AcOEt. M.p. 227°.

*Methiodide*: needles from EtOH.Aq. M.p. 255-6° (223°).

*Acetyl*: prisms from  $Et_2O$ -pet. ether. M.p. 100-1°. *Methiodide*: prisms from EtOH. M.p. 223-5°. *Semicarbazone*: needles from EtOH. M.p. 249°. *Phenylhydrazone*: prisms from EtOH. M.p. 225-6°.

*Picrate*: prisms from EtOH. M.p. 250-3°.

Schöpf, Hirsch, *Ann.*, 1931, 489, 240.

Gulland, *J. Chem. Soc.*, 1928, 702.

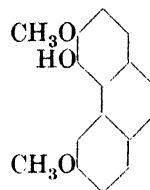
Schöpf, Borkowsky, *Ann.*, 1927, 458, 148.

Gulland, Robinson, *J. Chem. Soc.*, 1923, 998.

Pschorr, *Ber.*, 1905, 38, 3163.

### Thebaol anthrene)

(4-Hydroxy-3:6-dimethoxyphen-



$C_{16}H_{14}O_3$

MW, 254

Plates and columns from AcOH. M.p. 94°. Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $Me_2CO$ ,  $C_6H_6$ . Spar. sol. AcOH, ligroin. Zn dust dist.  $\rightarrow$  phenanthrene.

*Me ether*: methylthebaol.  $C_{17}H_{16}O_3$ . MW, 268. Thick oil. *Picrate*: reddish-brown needles from EtOH. M.p. 110-12°.

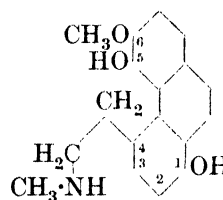
*Acetyl*: plates from EtOH. M.p. 118-22°. *Picrate*: red needles. M.p. 139°.

*Benzoyl*: needles from AcOH. M.p. 160-1°.

Pschorr, *Ber.*, 1912, 45, 2218.

Freund, Göbel, *Ber.*, 1897, 30, 1371, 1389.

### Thebenine (1:5-Dihydroxy-6-methoxy-4-[ $\beta$ -methylaminoethyl]-phenanthrene)



$C_{18}H_{19}O_3N$

MW, 297

Amorph. Spar. sol. boiling EtOH. Sol. aq. KOH. Insol.  $Et_2O$ ,  $C_6H_6$ . Sol. conc.  $H_2SO_4$   $\rightarrow$  blue col. Readily oxidises especially in presence of alkalis. Zn dust dist.  $\rightarrow$  pyrene.

*B,HCl*: plates +  $3H_2O$ . Sinters at 231°. M.p. 235°.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: yellow plates +  $1H_2O$ . Sinters at 205°. M.p. 209-10°.

*Oxalate*: prisms. M.p. 275-6°.

1-*Me ether*: methebenine.  $C_{19}H_{21}O_3N$ . MW, 311. Cryst. or amorph. from EtOH. Sinters at 155°. M.p. 165-7°. Pptd. from alk. sol. with  $CO_2$ . Hot dil. HCl  $\rightarrow$  thebenine. *B,HCl*: needles from EtOH. M.p. 250°. *B,HI*: plates from EtOH.Aq. Sinters at 190°. M.p. 195-8°. *Sulphate*: needles. M.p. 238.5°. N:5-*Di-acetyl*: plates from EtOH. M.p. 179°. N:5-*Dibenzoyl*: needles from EtOH. M.p. 159°.

N-*Me*: *methiodide*: thebeninmethine methiodide. Cryst. from EtOH. M.p. 206-8°.

N:1-*Di-Me*:  $C_{20}H_{23}O_3N$ . MW, 325. *Meth-*

*iodide*: methebeninmethin methiodide. Columns from EtOH.Aq. M.p. 215°.

*1:5-Di-Me ether*: *methiodide*: aggregates of needles from EtOH. M.p. 247°. *Methosulphate*: needles from EtOH or Et<sub>2</sub>O. Sinters at 268°. M.p. 277°.

*1-Et ether*: ethebenine. C<sub>20</sub>H<sub>23</sub>O<sub>3</sub>N. MW, 325. Yellow, amorph. *B,HCl*: microplates from EtOH.Aq. M.p. 248°. *B,HI*: plates + 1H<sub>2</sub>O from H<sub>2</sub>O. Sinters at 200°. M.p. 206-7°. *N:5-Diacetyl*: cryst. from EtOH. M.p. 163°.

*N-Me*: *1-Et*: C<sub>21</sub>H<sub>25</sub>O<sub>3</sub>N. MW, 339. *Methiodide*: ethebeninmethin methiodide. Plates from EtOH.Aq. Sinters at 210°. M.p. 215°.

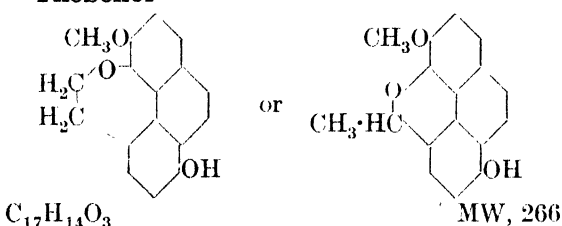
*1-Propyl ether*: prothebenine. C<sub>21</sub>H<sub>25</sub>O<sub>3</sub>N. MW, 339. Needles from EtOH. Sinters at 167°. M.p. 172-3°. *B,HCl*: plates from EtOH. Sinters at 215°. M.p. 221-2°. *B,HI*: plates. M.p. 212-13°.

*N:1:5-Triacetyl*: hydrated needles from EtOH.Aq. M.p. 72-80°. Anhyd. cryst. from abs. EtOH. M.p. 160-1°.

Gulland, Virden, *J. Chem. Soc.*, 1928, 921.  
Pschorr, *Ann.*, 1910, 373, 69.

Freund, Holthof, *Ber.*, 1899, 32, 179, 181.  
Freund, Michaels, *Ber.*, 1897, 30, 1375.

### Thebenol



Rhombohedral from hot AcOH. M.p. 186-8°. Very sol. EtOH, Et<sub>2</sub>O. Sol. C<sub>6</sub>H<sub>6</sub>, KOH.Aq. Insol. H<sub>2</sub>O, ligroin, Na<sub>2</sub>CO<sub>3</sub>, NH<sub>4</sub>OH. Alc. sol. reduces AgNO<sub>3</sub>. Zn dust dist., or heat with HI(+P) → pyrene. KOH fusion → northebenol.

*Na salt*: cryst. ppt. M.p. about 210-12°.

*Me ether*: methebenol. C<sub>18</sub>H<sub>16</sub>O<sub>3</sub>. MW, 280. Plates from AcOH. M.p. 133-4°. Very sol. CHCl<sub>3</sub>. *Picrate*: m.p. 106°.

*Et ether*: C<sub>19</sub>H<sub>18</sub>O<sub>3</sub>. MW, 294. Plates from AcOH. M.p. 104°. Spar. sol. EtOH, ligroin.

*Propyl ether*: C<sub>20</sub>H<sub>20</sub>O<sub>3</sub>. MW, 308. Plates from AcOH. M.p. 103-5°.

Gulland, Virden, *J. Chem. Soc.*, 1928, 921.

Sieglitz, Koch, *Ber.*, 1925, 58, 78.

Pschorr, *Ann.*, 1910, 373, 61.

Freund, Holthof, *Ber.*, 1899, 32, 184.

Freund, Michaels, *Ber.*, 1897, 30, 1380.

### Theelin.

See Estrone.

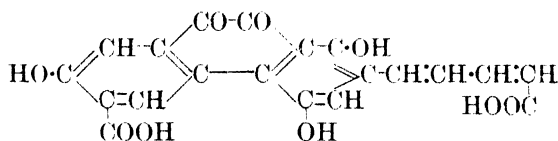
### Theelol.

See Estriol.

### Theine.

See Caffeine.

### Thelephoric Acid

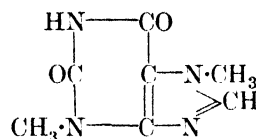


Isolated from *Thelephora palmata*. Black powder with metallic reflex. Almost insol. in all org. solvents except Py.

*Triacetyl*: orange-yellow needles from PhNO<sub>2</sub>. Decomp. at 330°.

Kögl, Erxleben, Jänecke, *Ann.*, 1930, 482, 110.

### Theobromine (3:7-Dimethylxanthine, 3:7-dimethyl-2:6-dihydroxypurine)



Important alkaloid constituent of cacao beans, kola nuts, etc. Rhombic microcryst. Sublimes at 290°. Insol. ligroin, cold Et<sub>2</sub>O, cold CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Sol. 1600 parts H<sub>2</sub>O at 17°, 3125 parts Et<sub>2</sub>O at b.p.  $k = 11.1 \times 10^{-11}$  at 25° ( $4.6 \times 10^{-14}$  at 40°). CrO<sub>3</sub> → methylparabanic acid + methylamine. Methylation → caffeine.

Faintly basic. Salts decomp. by H<sub>2</sub>O. Forms more stable salts with bases. Alkaloid and salts have diuretic action.

*Perchlorate*: cryst. + 1H<sub>2</sub>O. Decomp. at 271-3°.

*Hg salt*: darkens at 295-305°. M.p. 310°.

*B,HgNO<sub>3</sub>*: prisms. Does not melt below 300°.

*N-Me*: see Caffeine.

*N-Et*: C<sub>9</sub>H<sub>12</sub>O<sub>2</sub>N<sub>4</sub>. MW, 208. Needles from H<sub>2</sub>O. M.p. 164-5°. Sublimes. *B,HAuCl<sub>4</sub>*: needles. M.p. 226°.

*N-Propyl*: C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>N<sub>4</sub>. MW, 222. Needles from H<sub>2</sub>O. M.p. 136°. *B,HAuCl<sub>4</sub>*: needles. M.p. 95°.

*N-Butyl*: C<sub>11</sub>H<sub>16</sub>O<sub>2</sub>N<sub>4</sub>. MW, 236. Needles from H<sub>2</sub>O. M.p. 119°.

*N-Isobutyl*: m.p. 129-30°. *B,HAuCl<sub>4</sub>*: needles. M.p. 97°.



**Methochloride**: colourless columns. M.p. 320–40° decomp. Very sol. H<sub>2</sub>O.

**Methochloroaurate**: yellow woolly needles. M.p. 265° decomp.

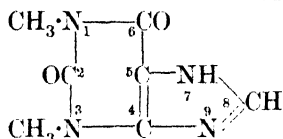
Biltz, Max, *Ann.*, 1921, **423**, 320.

Dubosc, *Chem. Zentr.*, 1932, IV, 956.

### Theocin.

See Theophylline.

**Theophylline** (*Theocin*, 1 : 3-dimethylxanthine, 1 : 3-dimethyl-2 : 6-dihydroxypurine)



C<sub>7</sub>H<sub>8</sub>O<sub>2</sub>N<sub>4</sub> MW, 180

Alkaloid constituent of tea leaves. Needles or plates + 1H<sub>2</sub>O from hot H<sub>2</sub>O. M.p. 264°. Very sol. warm H<sub>2</sub>O. Spar. sol. cold EtOH.  $k = 16.2 \times 10^{-10}$  at 25° ( $5.46 \times 10^{-14}$  at 40°). Methylation → caffeine. Alkaloid and metallic salts have diuretic action. Comps. with Na acetate and Na salicylate are important diuretics.

7-N-Me: see Caffeine.

7-N-Propyl: C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>N<sub>4</sub>. MW, 222.

B,HAuCl<sub>4</sub>: yellow needles + 2H<sub>2</sub>O. M.p. 214°.

7-N-Isopropyl: B,HAuCl<sub>4</sub>: yellow needles from EtOH.Aq. M.p. 183°. B,H<sub>2</sub>PtCl<sub>6</sub>: orange-yellow cryst. + 2H<sub>2</sub>O. M.p. 201°.

7-N-Acetyl: needles from CHCl<sub>3</sub>. M.p. 158°.

7-N-Benzoyl: needles from EtOH. M.p. 202°.

Yoshitomi, *Chem. Abstracts*, 1925, **19**, 2303.

Biltz, Strufe, *Ann.*, 1914, **404**, 137, 170.

Schwabe, *Arch. pharm.*, 1907, **245**, 312.

Fischer, *Ber.*, 1897, **30**, 553.

### Thermopsine

C<sub>15</sub>H<sub>20</sub>ON<sub>2</sub> MW, 244

Alkaloid isolated from *Thermopsis lanceolata*. Pale yellow cryst. from Me<sub>2</sub>CO. M.p. 206–206.5° (203–5°). Sol. H<sub>2</sub>O, Et<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Spar. sol. Me<sub>2</sub>CO, pet. ether.  $[\alpha]_D^{20} = 159.6^\circ$  in EtOH.

B,HCl: m.p. 247–8° decomp.

B,HI: needles from EtOH. M.p. 294–6° (306–8° decomp.).

Chloroaurate: m.p. 183–4° decomp.

Chloroplatinate: orange-red prisms. Decomp. at 245–7° (254–6°).

Methiodide: prisms from MeOH. M.p. 241–2° decomp.

Picrate: yellow prisms from EtOH-Me<sub>2</sub>CO. M.p. 229–31° decomp. (208–9°).

**Picrolonate**: m.p. 173–5° decomp.

Orechoff, *Chem. Zentr.*, 1935, II, 2215.

Orechoff, Gurewitsch, *Ber.*, 1935, **68**, 820.

Orechoff, Norkina, Gurewitsch, *Ber.*, 1933, **66**, 627.

### Thevetin

C<sub>42</sub>H<sub>66</sub>O<sub>18</sub> MW, 858

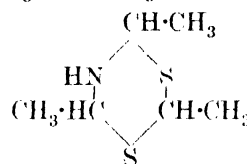
Glucoside from leaves of *Thevetia nereifolia*, Juss., etc. Needles from EtOH or isopropyl alcohol. Softens at 193–4°. M.p. 210°. Sol. EtOH, C<sub>3</sub>H<sub>7</sub>OH, Py. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Gives positive Legal test.  $[\alpha]_D^{25} = 62.5^\circ$ . Hyd. → thevetigenin + glucose + digitalose (?).

Tschesche, *Ber.*, 1936, **69**, 2368.

Elderfield, *J. Biol. Chem.*, 1936, **115**, 247.

Chen, Chen, *J. Biol. Chem.*, 1934, **105**, 231.

**Thialdine** (Thioacetaldehyde-ammonia, 2 : 4 : 6-trimethyl-5 : 6-dihydro-1 : 3 : 5-dithiazine)



C<sub>6</sub>H<sub>13</sub>NS<sub>2</sub> MW, 163

Cryst. M.p. 43°. Very sol. Et<sub>2</sub>O. Sol. EtOH. Spar. sol. H<sub>2</sub>O. Volatile in steam. Forms cryst. salts with acids.

B,HCNS: needles. M.p. 132° decomp.

N-Me: C<sub>7</sub>H<sub>15</sub>NS<sub>2</sub>. MW, 177. Needles from EtOH. M.p. 79°. Very sol. Et<sub>2</sub>O. Spar. sol. cold EtOH. Insol. cold H<sub>2</sub>O. B,HCNS: columns from EtOH. M.p. 120°.

Marckwald, *Ber.*, 1886, **19**, 2381.

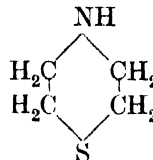
### Thianthrene.

See Diphenylene disulphide.

### Thiasine.

See Ergothioneine.

**1 : 4-Thiazan** (Tetrahydrothiazine, thiomorpholine)



C<sub>4</sub>H<sub>8</sub>NS MW, 103

Colourless liq. B.p. 169°. Misc. with H<sub>2</sub>O and most org. solvents.

B,HCl: needles from C<sub>6</sub>H<sub>6</sub>. Softens at 145°. M.p. 160–5°.

*Chloroplatinate*: does not melt below 250°.

*N-Me*:  $C_5H_{11}NS$ . MW, 117. B.p. 163-4°. Misc. with  $H_2O$ .  $D_4^{20}$  0.9924.  $n_D^{20}$  1.50176. *B,HCl*: m.p. 239°. *Picrate*: yellow needles from EtOH. M.p. 226° decomp.

*N-Et*:  $C_6H_{13}NS$ . MW, 131. B.p. 184°. Misc. with  $H_2O$ .  $D_4^{20}$  0.98854.  $n_D^{20}$  1.50180. *B,HCl*: m.p. 188°. *Chloroplatinate*: decomp. at 222°. *Picrate*: yellow needles from  $Me_2CO$ -EtOH. M.p. 185-6°.

*N-Phenyl*:  $C_{10}H_{13}NS$ . MW, 179. Cryst. from toluene. M.p. 108-11°.

*Ethiodide*: cryst. from EtOH. M.p. 260°.

*Picrate*: orange needles from EtOH. M.p. 198° decomp.

*Picrolonate*: orange prisms from EtOH. M.p. 242° decomp.

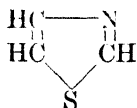
Lawson, Reid, *J. Am. Chem. Soc.*, 1925, **47**, 2830.

Helfrich, Reid, *J. Am. Chem. Soc.*, 1920, **42**, 1226.

Davies, *J. Chem. Soc.*, 1920, **117**, 396.

Clarke, *J. Chem. Soc.*, 1912, **101**, 1586.

#### Thiazole



$C_3H_3NS$

MW, 85

Liq. B.p. 116-8°.  $D_4^{17}$  1.1998.

*B,HgCl<sub>2</sub>*: cryst. ppt. Softens at 200°. M.p. 225° decomp.

*B,HgCl<sub>2</sub>,HCl*: needles. M.p. 103-4°.

*B<sub>2</sub>H<sub>2</sub>PiCl<sub>6</sub>*: prisms + 2 $H_2O$ . Decomp. at 250°.

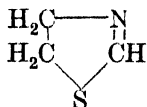
*B,HAuCl<sub>4</sub>*: yellow prisms from EtOH. M.p. 258° decomp.

*Picrate*: needles. Sinters at 150°. M.p. 159-60° decomp.

Gabriel, Bachstsz, *Ber.*, 1914, **47**, 3169.

Popp, *Ann.*, 1889, **250**, 275.

#### 4 : 5-Thiazoline (4 : 5-Dihydrothiazole)



$C_3H_5NS$

MW, 87

B.p. 137.5-138°/747 mm.

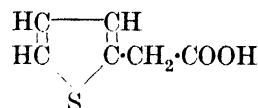
*Picrate*: yellow plates. M.p. 150-1°.

Gabriel, *Ber.*, 1916, **49**, 1111.

#### Thienone.

See Dithienyl Ketone.

#### 2-Thienylacetic Acid (*Thiophene-2-acetic acid*)



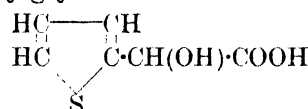
$C_6H_6O_2S$

MW, 142

Cryst. M.p. 76°. Sol. hot  $H_2O$ , EtOH, Et<sub>2</sub>O.

Ernst, *Ber.*, 1886, **19**, 3281.

#### 2-Thienylglycollic Acid



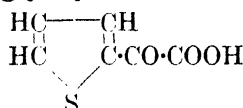
$C_6H_6O_3S$

MW, 158

Needles from  $C_6H_6$ . M.p. 115°. Very sol.  $H_2O$ , EtOH, Et<sub>2</sub>O. Red.  $\rightarrow$  thienylacetic acid.  $MnO_2 + H_2O \rightarrow$  thiophene-2-aldehyde.

See previous reference.

#### 2-Thienylglyoxylic Acid



$C_6H_4O_3S$

MW, 156

Cryst. + 1 $H_2O$ . M.p. 58-9°, anhyd. 91.5°. Very sol.  $H_2O$ . Very strong acid. Heat  $\rightarrow$  thiophene-2-aldehyde. Ox.  $\rightarrow$  thiophene-2-carboxylic acid. AcOH sol. + AcOH- $H_2SO_4$   $\rightarrow$  brown  $\rightarrow$  green  $\rightarrow$  violet  $\rightarrow$  blue col. *Me ester*:  $C_7H_6O_3S$ . MW, 170. M.p. 28.5°. *Oxime*: needles from EtOH.Aq. Softens at 97°. M.p. 104-5°.

*Et ester*:  $C_8H_8O_3S$ . MW, 184. Oil with fragrant odour. B.p. 264-5° slight decomp. *Oxime*: needles. M.p. 122-3°.

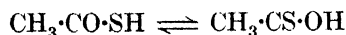
*Amide*:  $C_6H_5O_2NS$ . MW, 155. Needles. M.p. 88°. Very sol. EtOH, Et<sub>2</sub>O. Insol.  $H_2O$ . *Oxime*: needles. M.p. 145-6° decomp. *Acetyl*: prisms. M.p. 85-7° decomp.

*Phenyldiazone*: yellow needles from Et<sub>2</sub>O. M.p. 164-5° decomp.

Bradley, *Ber.*, 1886, **19**, 2116.

Peter, *Ber.*, 1885, **18**, 537.

#### Thioacetic Acid



$C_2H_4OS$

MW, 76

B.p. 93°.  $D_4^{10}$  1.075. Very sol. EtOH. Sol.  $H_2O$ .  $k = 4.69 \times 10^{-4}$  at 25°.

*Bi salt*: prisms. M.p. 85°.

*S-Me ester*:  $C_3H_6OS$ . MW, 90. B.p. 95-6°.

*O-Me ester*: pale yellow liq. B.p. 88–91°.  $D_4^{25}$  0.9002.  $n_D^{25}$  1.4212. Insol.  $H_2O$ .

*S-Et ester*:  $C_4H_8OS$ . MW, 104. B.p. 116–17°.  $D_4^{25}$  0.9755.  $n_D^{25}$  1.4503.

*O-Et ester*: b.p. 105–7°.  $D_4^{17}$  0.8980.

*S-Propyl ester*:  $C_5H_{10}OS$ . MW, 118. B.p. 135–7°.

*O-Propyl ester*: b.p. 125–30°.  $D_4^{25}$  0.8952.  $n_D^{25}$  1.4283.

*S-Isopropyl ester*: b.p. 124–7°.

*O-Isopropyl ester*: b.p. 119–22°.  $D_4^{22}$  0.8901.

*O-Butyl ester*:  $C_6H_{12}OS$ . MW, 132. B.p. 146–9°.  $D_4^{25}$  0.8883.  $n_D^{22}$  1.4501,  $n_D^{25}$  1.4196.

*O-Isobutyl ester*: b.p. 135–40°.  $D_4^{26}$  0.8875.  $n_D^{26}$  1.4316.

*S-Phenyl ester*:  $C_8H_8OS$ . MW, 152. B.p. 228–30°.  $D_4^{23}$  1.117.  $n_D^{22}$  1.5706.

*O-Phenyl ester*: b.p. 90–4°/38 mm.  $D_4^{20}$  0.9914.

*O-Benzyl ester*:  $C_9H_{10}OS$ . MW, 166. B.p. 115–20°/27–9 mm.  $D_4^{25}$  1.0296.  $n_D^{26}$  1.5492.

*Anhydride*: see Diacetyl sulphide.

*Amide*: thioacetamide.  $C_3H_5NS$ . MW, 75. Prisms from  $Et_2O$ . M.p. 107.5–108.5° (115°). Sol.  $H_2O$ ,  $EtOH$ . Spar. sol.  $Et_2O$ .

*Hydrazide*: m.p. 59°.

*Anilide*: thioacetanilide. Needles from  $H_2O$ . M.p. 75–6°.

*o-Toluidide*: needles from ligroin. M.p. 91–2°.

*m-Toluidide*: needles. M.p. 42–3°.

*p-Toluidide*: prisms from  $EtOH$ . Aq. M.p. 129.5–130.5°.

*o-Aniside*: yellow plates. M.p. 52–3°.

*p-Aniside*: needles. M.p. 114°.

Baker, Reid, *J. Am. Chem. Soc.*, 1929, **51**, 1568.

Sakurada, *Brit. Chem. Abstracts*, 1927, **133**.

Clarke, Hartman, *J. Am. Chem. Soc.*, 1924, **46**, 1732.

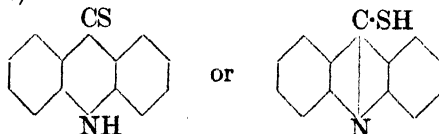
Delépine, *Compt. rend.*, 1911, **153**, 281.

Schoff, *Ber.*, 1895, **28**, 1205.

### Thioacridol.

See Thioacridone.

**Thioacridone** (9-Mercaptoacridine, thioacridol)



$C_{13}H_9NS$

MW, 211

Brownish-yellow needles +  $1H_2O$  from 2%  $NaOH$ . M.p. 275°. Loses  $H_2O$  at 120° over  $H_2SO_4$ . Sol.  $Me_2CO$ . Spar. sol. other org. sol-

vents. Sol. conc. acids, alkalis and  $NH_4OH$ . Insol. alkali carbonates. Salts are unstable and decomp. by  $H_2O$ .

*S-Me*:  $C_{14}H_{11}NS$ . MW, 225. Greenish-yellow needles from  $EtOH$ . M.p. 113–14°. *Hydrochloride*: yellow needles. M.p. 198°. *Sulphate*: m.p. 156–7°. *Nitrate*: m.p. 117–18°. *Picrate*: needles from  $EtOH-CHCl_3$ . M.p. 205°.

*S-Et*:  $C_{15}H_{13}NS$ . MW, 239. Yellow needles from pet. ether. M.p. 65°. *Picrate*: yellow needles from  $CHCl_3-EtOH$ . M.p. 182–3°.

*N-Phenyl*:  $C_{19}H_{13}NS$ . MW, 287. Red cryst. from  $AcOH$ . M.p. 227–8°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol.  $EtOH$ ,  $AcOH$ .

*S-2:4-Dinitrophenyl*: yellow needles from xylene. M.p. 290° decomp. *Picrate*: yellow needles. M.p. 226° decomp.

*S-2:4:6-Trinitrophenyl*: thioacridol picryl ether. Red needles from  $CHCl_3$ . M.p. 233° decomp.

*S-Benzyl*: needles from  $EtOH$ . Aq. M.p. 109°. *Hydrochloride*: yellow leaflets. Decomp. at 140–1°. *Sulphate*: m.p. 179–80° decomp. *Nitrate*: yellow leaflets. M.p. 106–7° decomp. *Picrate*: yellowish-brown needles from  $EtOH$ . M.p. 189–90°.

*S-o-Nitrobenzyl*: yellow leaflets. M.p. 129–30°. *Picrate*: yellow cryst. M.p. 190–1°.

*S-p-Nitrobenzyl*: yellow cryst. from  $Me_2CO$ . M.p. 152°. *Picrate*: m.p. 204°.

*S-Benzoyl*: yellow leaflets from  $EtOH-CHCl_3$ . M.p. 209°. *Picrate*: greenish-yellow needles from  $EtOH-CHCl_3$ . M.p. 190°.

Edinger, *J. prakt. Chem.*, 1903, **68**, 88.

Edinger, Arnold, *J. prakt. Chem.*, 1901, **64**, 196, 487.

Kalle, D.R.P., 120,586, (*Chem. Zentr.*, 1901, **I**, 1254).

Schönberg, Schütz, Nickel, *Ber.*, 1928, **61**, 1383.

### Thioaniline.

See 4:4'-Diaminodiphenyl sulphide.

**Thioanisaldehyde** (*p*-Methylmercaptobenzaldehyde)



$C_8H_8OS$

MW, 152

Yellowish plates from ligroin. M.p. 78°. B.p. 273°. Sol. common org. solvents.

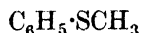
*Phenylhydrazone*: m.p. 138° (136°).

Friedländer, Lenk, *Ber.*, 1912, **45**, 2089.

Mitra, *Chem. Abstracts*, 1933, **27**, 3923.

**Thioanisidine.**

See under Amino thiophenol.

**Thioanisole** (*Methyl phenyl sulphide*)
 $\text{C}_7\text{H}_8\text{S}$  MW, 124

 B.p. 187–8°, 58–60°/6 mm.  $D_4^{25}$  1.0533.  $n_D^{25}$  1.5832.

 Knapp, *Monatsh.*, 1930, **56**, 68 (Footnote).  
 Suter, Hansen, *J. Am. Chem. Soc.*, 1932,  
**54**, 4101.

 Brand, Kranz, *J. prakt. Chem.*, 1927, **115**,  
 143.
**Thioanisole- $\omega$ -carboxylic Acid.**

See Phenylthioglycollic Acid.

**Thioanthrol.**

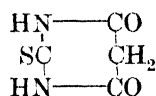
See Mercaptoanthracene.

**Thioargyrium.**

See under Dithiosalicylic Acid.

**Thioaspirin.**

See under Thiosalicylic Acid.

**Thiobarbituric Acid** (*Malonylthiourea*)
 $\text{C}_4\text{H}_4\text{O}_2\text{N}_2\text{S}$  MW, 144

 Plates from  $\text{H}_2\text{O}$ . M.p. 235° decomp. (rapid  
 heat.).

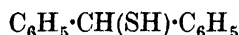
 Michael, *J. prakt. Chem.*, 1894, **49**, 38.

 Harwood, *Chem. Abstracts*, 1933, **27**, 1676.
**Thiobenzamide.**

See under Thiobenzoic Acid.

**Thiobenzanilide.**

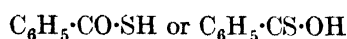
See under Thiobenzoic Acid.

**Thiobenzhydrol** (*Benzhydryl mercaptan*,  
 $\alpha$ -mercaptodiphenylmethane)

 $\text{C}_{13}\text{H}_{12}\text{S}$  MW, 200

Yellow oil. B.p. 128–30°/1.2 mm.

 Staudinger, Siegwart, *Ber.*, 1916, **49**, 1920.
**Thiobenzilic Acid** ( $\alpha$ -*Mercaptodiphenylacetic acid*)
 $\text{C}_{14}\text{H}_{12}\text{O}_2\text{S}$  MW, 244

 Plates from  $\text{AcOH}$ . M.p. 147.5–149°. Sol.  
 $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ , boiling  
 $\text{MeOH}$ . Very spar. sol. boiling  $\text{H}_2\text{O}$ .

 Bettschart, Bistrzycki, *Helv. Chim. Acta*,  
 1919, **2**, 127.
**Thiobenzoic Acid**
 $\text{C}_7\text{H}_6\text{OS}$  MW, 138

 Yellow oil. Solidifies in ice and then melts  
 about 24°. Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ . Insol.  
 $\text{H}_2\text{O}$ . Volatile in steam. Decomp. on dist.

 $\text{NH}_4$  salt: cryst. M.p. 118°. Sol.  $\text{H}_2\text{O}$ .

 $\text{O-Me ester}$ :  $\text{C}_8\text{H}_8\text{OS}$ . MW, 152. Yellow  
 liq. B.p. 110–12°/10 mm. Insol.  $\text{H}_2\text{O}$ . Fumes  
 in air.

 $\text{S-Me ester}$ : b.p. 231–2°, 134°/25 mm.  $D_{25}^{25}$   
 1.1381.

 $\text{S-Et ester}$ :  $\text{C}_9\text{H}_{10}\text{OS}$ . MW, 166. B.p. 252–3°,  
 146°/31 mm.  $D_{25}^{25}$  1.0977.

 $\text{S-Propyl ester}$ :  $\text{C}_{10}\text{H}_{12}\text{OS}$ . MW, 180. B.p.  
 251–5°, 144°/13 mm.  $D_{25}^{25}$  1.0724.

 $\text{S-Butyl ester}$ :  $\text{C}_{11}\text{H}_{14}\text{OS}$ . MW, 194. B.p.  
 160°/23 mm.  $D_{25}^{25}$  1.0514.

 $\text{S-sec.-n-Butyl ester}$ : b.p. 151°/23 mm.  $D_{25}^{25}$   
 1.0488.

 $\text{S-Isobutyl ester}$ : b.p. 150°/20 mm.  $D_{25}^{25}$  1.0457.

 $\text{S-tert.-Butyl ester}$ : b.p. 110°/28 mm.  $D_{25}^{25}$   
 1.0468.

 $\text{S-Phenyl ester}$ :  $\text{C}_{13}\text{H}_{10}\text{OS}$ . MW, 214. Needles  
 from  $\text{EtOH}$  or  $\text{C}_6\text{H}_6$ . M.p. 56°.  $n_D^{56.5}$  1.6231.  
 Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ .  $\text{KOH} \rightarrow$  thio-  
 phenol + benzoic acid.

 $\text{S-p-Tolyl ester}$ : cryst. from  $\text{EtOH}$ . M.p. 75°.

 $\text{S-Benzyl ester}$ : cryst. from  $\text{EtOH}$ . M.p.  
 39.5°.

 $\text{Amide}$ : thiobenzamide.  $\text{C}_7\text{H}_7\text{NS}$ . MW, 137.  
 Needles. M.p. 115–16°.  $\text{N-Benzoyl}$ : red cryst.  
 M.p. 117°.

 $\text{Methylamide}$ :  $\text{C}_8\text{H}_9\text{NS}$ . MW, 151. Yellow  
 needles from  $\text{EtOH}$ . M.p. 79°.

 $\text{Anilide}$ : thiobenzanilide. Yellow plates or  
 prisms. M.p. 101.5–102° (96°).

 $\text{N-Me-anilide}$ : m.p. 63° (59°). B.p. 331–2°.

 $\text{o-Toluidide}$ : m.p. 85–6°.

 $\text{p-Toluidide}$ : yellow needles from  $\text{EtOH.Aq.}$   
 M.p. 128.5–129.5°.

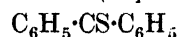
 $\text{Piperidide}$ : yellow plates from  $\text{MeOH.Aq.}$   
 M.p. 63–4°. B.p. 205°/12 mm.

 $\text{Anhydride}$ : see Dibenzoyl sulphide.

 Engelhardt, Latschinow, Malyschew, *Z.*  
*Chem.*, 1868, 354.

 Kym, *Ber.*, 1899, **32**, 3533 (Note).

 Hantzsch, Scharf, *Ber.*, 1913, **46**, 3584.

 Gabriel, Heymann, *Ber.*, 1890, **23**, 158.
**Thiobenzophenone** (*Diphenyl thioether*)
 $\text{C}_{13}\text{H}_{10}\text{S}$  MW, 198

 Blue needles from pet. ether. M.p. 53–4°.  
 B.p. 174°/14 mm. Very sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

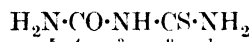
Spar. sol. pet. ether, cold EtOH. Standing in air  $\rightarrow$  benzophenone.

Staudinger, Freudenberger, *Organic Syntheses*, 1931, XI, 94.

### Thiobenzyl Alcohol.

See Benzyl Mercaptan.

### Thiobiuret



$\text{C}_2\text{H}_5\text{ON}_3\text{S}$  MW, 119

Needles +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. anhyd.  $186^\circ$ . Sol. EtOH, AcOH. Mod. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{CS}_2$ , ligroin,  $\text{C}_6\text{H}_6$ . Sol. alkalis.

5-Phenyl: cryst. from EtOH. M.p.  $186^\circ$ .

1:5-Diphenyl: m.p.  $161^\circ$ .

Wunderlich, *Ber.*, 1886, **19**, 452.

Hecht, *Ber.*, 1892, **25**, 749.

### Thiobutyric Acid

$\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CO}\cdot\text{SH}$  or  $\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CS}\cdot\text{OH}$   
 $\text{C}_4\text{H}_8\text{OS}$  MW, 104

B.p.  $130^\circ$ . Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ .

S-Phenyl ester:  $\text{C}_{10}\text{H}_{12}\text{OS}$ . MW, 180. Yellow liq. B.p.  $210\text{--}12^\circ/20$  mm. Volatile in steam.

Anilide: needles from AcOH.Aq. M.p.  $32\text{--}3^\circ$ .

Ulrich, *Ann.*, 1859, **109**, 280.

### Thiocarbamic Acid

$\text{H}_2\text{N}\cdot\text{CO}\cdot\text{SH}$  or  $\text{H}_2\text{N}\cdot\text{CS}\cdot\text{OH}$   
 $\text{CH}_3\text{ONS}$  MW, 77

Known only in form of its salts and esters.

$\text{NH}_4$  salt: colourless cryst. Very sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ . Dry salt rapidly turns yellow in air  $\rightarrow (\text{NH}_4)_2\text{S}$ .

S-Me ester:  $\text{C}_2\text{H}_5\text{ONS}$ . MW, 91. Prisms from  $\text{Et}_2\text{O}$ . M.p.  $107\text{--}8^\circ$  ( $95\text{--}8^\circ$ ). N-Acetyl: needles from  $\text{C}_6\text{H}_6$ . M.p.  $145.5\text{--}146^\circ$ .

O-Me ester: m.p.  $43^\circ$ . N-Acetyl: cryst. from pet. ether. M.p.  $79\text{--}80^\circ$ .

Et ester: see Thiourethane.

O-Propyl ester:  $\text{C}_4\text{H}_9\text{ONS}$ . MW, 119. Cryst. M.p.  $35^\circ$ .

S-Isopropyl ester: plates. M.p.  $125^\circ$ .

S-Isobutyl ester:  $\text{C}_5\text{H}_{11}\text{ONS}$ . MW, 133. Plates from  $\text{H}_2\text{O}$ . M.p.  $102\text{--}3^\circ$ . Volatile in steam.

O-Isobutyl ester: plates from  $\text{H}_2\text{O}$ . M.p.  $51\text{--}3^\circ$  ( $36^\circ$ ).

S-Phenyl ester:  $\text{C}_7\text{H}_7\text{ONS}$ . MW, 153. Plates from  $\text{C}_6\text{H}_6$  or  $\text{H}_2\text{O}$ . M.p.  $96\text{--}8^\circ$ .

O-Phenyl ester: needles from EtOH. M.p.  $132\text{--}132.5^\circ$ .

S-Benzyl ester:  $\text{C}_8\text{H}_9\text{ONS}$ . MW, 167. Plates from  $\text{C}_6\text{H}_6$ . M.p.  $125^\circ$ .

Amide: see Thiourea.

Wheeler, Barnes, *Am. Chem. J.*, 1899, **22**, 146.

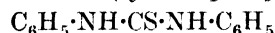
Kretzschmar, *J. prakt. Chem.*, 1873, **7**, 474.

Salomon, *J. prakt. Chem.*, 1873, **8**, 115.

### Thiocarbamide.

See Thiourea.

Thiocarbamilide (sym.-Diphenylthiourea)



$\text{C}_{13}\text{H}_{12}\text{N}_2\text{S}$  MW, 228

Leaflets from EtOH. M.p.  $154\text{--}5^\circ$  ( $150.5^\circ$ ). Sol. EtOH,  $\text{Et}_2\text{O}$ . Mod. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{CS}_2$ . Sol. alkalis, re-ppd. by acids. Triboluminescent. Very bitter taste.

$\text{B}_2\text{HgCl}_2$ : m.p.  $85^\circ$ .

$\text{B}_2\text{HgI}_2$ : m.p.  $183^\circ$ .

$\text{B}_2\text{HgI}_2$ : yellow prisms. M.p.  $139^\circ$ .

$\text{B}_2\text{AuCl}_3$ : prisms. M.p.  $194^\circ$ .

$\text{B}_2\text{CuBr}_2$ : prisms. M.p.  $187^\circ$ .

$\text{B}_2\text{CdBr}_2$ : m.p.  $140^\circ$ .

$\text{B}_2\text{ZnCl}_2$ : prisms. M.p.  $172^\circ$ .

$\text{B}_2\text{SnCl}_4$ : prisms. M.p.  $260^\circ$ .

Fry, *J. Am. Chem. Soc.*, 1913, **35**, 1541.

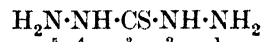
Drozдов, *Chem. Abstracts*, 1932, **26**, 5293.

Mistry, Guha, *Chem. Abstracts*, 1931, **25**, 1504.

Silesia Verein, D.R.P., 559,814, (*Chem. Zentr.*, 1933, I, 2463).

Rubber Service Labs., U.S.P., 1,688,707, (*Chem. Abstracts*, 1929, **23**, 156).

Thiocarbazine (Thiocarbohydrazide, dihydrazide of thiocarbonic acid)



$\text{CH}_6\text{N}_4\text{S}$  MW, 106

Cryst. from  $\text{H}_2\text{O}$ . Decomp. at  $170^\circ$ .

1:5-Diacetyl: hygroscopic cryst. M.p.  $204\text{--}5^\circ$ .

1-Phenyl:  $\text{C}_7\text{H}_{10}\text{N}_4\text{S}$ . MW, 182. Tables from EtOH. M.p.  $149\text{--}50^\circ$ . Sol. EtOH, AcOH, Py, alkalis. Spar. sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , HCl.  $\text{B}_2\text{HCl}$ : plates. M.p.  $181^\circ$ .

1-Me-1-Phenyl:  $\text{C}_8\text{H}_{12}\text{N}_4\text{S}$ . MW, 196. Prisms from EtOH. M.p.  $228\text{--}9^\circ$  decomp.

Guha, Roy-Choudhury, *J. Indian Chem. Soc.*, 1928, **5**, 149.

Guha, De, *J. Chem. Soc.*, 1924, 1215.

### Thiocarbonic Acid

$\text{HO}\cdot\text{CO}\cdot\text{SH}$  or  $\text{HO}\cdot\text{CS}\cdot\text{OH}$   
 $\text{CH}_2\text{O}_2\text{S}$  MW, 78

Does not exist in free state.

O:S-Di-Me ester:  $\text{C}_3\text{H}_6\text{O}_2\text{S}$ . MW, 106. Oil

with ethereal odour. B.p. 120–1°.  $D_4^{21}$  1.1203.  $n_D^{23}$  1.4524.

O-Di-Me ester: b.p. 119–20°.  $D_4^{21}$  1.1028.  $n_D^{24}$  1.4596.

O-S-Di-Et ester:  $C_5H_{10}O_2S$ . MW, 134. B.p. 156°.  $D_4^{18}$  1.0285.

O-Di-Et ester: b.p. 158–9°.  $D_4^{17.5}$  1.0267.  $n_D^{17.5}$  1.4601.

O:S-Diphenyl ester:  $C_{13}H_{10}O_2S$ . MW, 230. Needles from EtOH. M.p. 56°.

O-Diphenyl ester: plates from EtOH. M.p. 106°. Sol. most ord. org. solvents. Insol.  $H_2O$ .

Anhydride: see Carbonyl sulphide.

Amide: see Thiocarbamic Acid.

Chloride: see Thiocarbonyl chloride.

Hydrazide: see Thiocarbazine.

Nitrile: see Thiocyanic acid.

Anil: see Phenyl isothiocyanate.

Delépine, *Ann. chim.*, 1912, **25**, 547;  
*Compt. rend.*, 1910, **150**, 878; *Bull. soc. chim.*, 1910, **7**, 409, 727.

### Thiocarbonyl chloride (*Thiophosgene*)



MW, 115

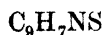
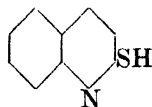
Red liq. with acrid odour. B.p. 73–6°. Fumes in air.  $D_4^{16}$  1.5085.  $n_D^{20}$  1.5442. When pure is not decomp. by light. Slowly decomp. by cold  $H_2O$ , rapidly with hot EtOH or NaOEt → esters.

Dyson, *Organic Syntheses*, 1926, VI, 86.

### Thiocarbonyl tetrachloride.

See Perchloromethyl Mercaptan.

**Thiocarbostyryl** (2-Mercaptoquinoline, 2-quinolyl mercaptan)



MW, 161

Yellow plates from EtOH.Aq. M.p. 175°. Very sol. hot EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Insol. cold  $H_2O$ . Sol. acids, alkalis.

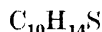
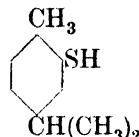
S-Me:  $C_{10}H_9NS$ . MW, 175. *Methiodide*: yellow cryst. from EtOH. M.p. 189°.

N-Me: greenish-yellow needles or prisms from EtOH. M.p. 118°. Spar. sol. hot  $H_2O$ .

S-Et:  $C_{11}H_{11}NS$ . MW, 189. Liq. Decomp. on dist. *B.H.I.*: yellow cryst. from EtOH- $Et_2O$ . M.p. 154°.  $B_2H_2PtCl_6$ : cryst. +  $H_2O$ . M.p. 190°. decomp.

Fischer, *Ber.*, 1899, **32**, 1305.

**Thiocarvacrol** (2-Mercapto-p-cymene, 2-methyl-5-isopropylthiophenol)



MW, 166

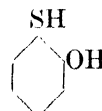
Colourless liq. with aromatic odour. B.p. 235–6°.  $D_4^{17.5}$  0.9975. Misc. with EtOH. Insol.  $H_2O$ .

*Hg salt*: needles. M.p. 109°.

S-Me:  $C_{11}H_{16}S$ . MW, 180. Liq. with unpleasant odour. B.p. 244°.

Kekulé, Fleischer, *Ber.*, 1873, **6**, 1088.

**Thiocatechol** (2-Hydroxythiophenol, o-hydroxyphenyl mercaptan)



MW, 126

Oil with strong odour. M.p. 5–6°. B.p. 216–17°/750.7 mm.  $D_4^0$  1.2373. Sol.  $Et_2O$ . Spar. sol.  $H_2O$ . Readily oxidises in alk. sol. to disulphide.

O-Me ether: o-methoxythiophenol, o-mercaptanisole, thioguaiacol.  $C_7H_8OS$ . MW, 140. Liq. B.p. 218–19°.

O-Et ether: o-mercaptophenetole.  $C_8H_{10}OS$ . MW, 154. Liq. B.p. 226–7°.

Di-Me ether: o-methoxythioanisole.  $C_8H_{10}OS$ . MW, 154. Liq. B.p. 237°.

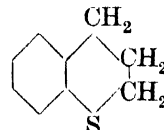
Di-Et ether: o-ethoxythiophenetole.  $C_{10}H_{14}OS$ . MW, 182. Liq. B.p. 248–50°.

Friedländer, Mauthner, *Chem. Zentr.*, 1904, II, 1176.

Gattermann, *Ber.*, 1899, **32**, 1147.

Haitinger, *Monatsh.*, 1883, **4**, 170.

**Thiochroman** (*Dihydrobenzthiopyran*)



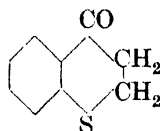
MW, 150

Yellow liq. B.p. 128–30°/15 mm. Volatile in steam.  $KMnO_4$  → thiochroman-S-dioxide.

S-Dioxide:  $C_9H_{10}O_2S$ . MW, 182. Cryst. from hot  $H_2O$ . M.p. 88.5°. Sol.  $H_2O$ . Very sol.  $Et_2O$ .

v. Braun, *Ber.*, 1910, **43**, 3225.

## Thiochromanone

C<sub>9</sub>H<sub>8</sub>OS

MW, 164

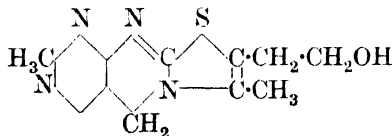
Colourless leaflets with mint-like odour from pet. ether. M.p. 29–30°. B.p. 154°/12 mm. H<sub>2</sub>SO<sub>4</sub> → red col.

*Oxime*: plates from EtOH–petrol. M.p. 98–100°.

*Semicarbazone*: needles from EtOH. M.p. 219–20°.

Krollpfeiffer, Schultze, *Ber.*, 1923, 56, 1822.

## Thiochrome

C<sub>12</sub>H<sub>14</sub>ON<sub>2</sub>S

MW, 262

The colouring matter of yeast. Yellow prisms from CHCl<sub>3</sub>. M.p. 227–8°. Sol. MeOH. Mod. sol. H<sub>2</sub>O. Spar. sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>. Gives blue fluor. in neutral or alk. sol. Sublimes in high vacuum. Has no physiological activity.

Todd, Bergel, *J. Chem. Soc.*, 1936, 1560.

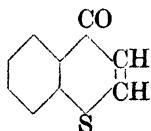
Todd, Bergel, Fraenkel-Conrat, Jacob, *J. Chem. Soc.*, 1936, 1601.

Kuhn, Vetter, *Ber.*, 1935, 68, 2375.

Barger, Bergel, Todd, *ibid.*, 2257.

Kuhn, Wagner-Jauregg, Klaveren, Vetter, *Z. physiol. Chem.*, 1935, 234, 196.

## Thiochromone (1 : 4-Benzthiopyrone)

C<sub>9</sub>H<sub>6</sub>OS

MW, 162

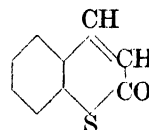
Colourless needles from ligroin. M.p. 78°.

Arndt *et al.*, *Ber.*, 1925, 58, 1620.

## Thiocoumarandione.

See Thionaphthenequinone.

## 1 - Thiocoumarin (o - Mercaptocinnamic lactam)

C<sub>9</sub>H<sub>6</sub>OS

MW, 162

Needles from ligroin. M.p. 80–80.5°. Spar. sol. H<sub>2</sub>O. Volatile in steam.

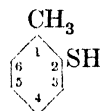
*Phenylhydrazine*: yellow plates from EtOH. M.p. 140°.

Simonis, Elias, *Ber.*, 1916, 49, 765.

## Thiocoumarone.

See Thionaphthene.

## o-Thiocresol (o-Mercaptotoluene)

C<sub>7</sub>H<sub>8</sub>S

MW, 124

Plates. M.p. 15°. B.p. 194.3°, 124.7°/100 mm., 106°/50 mm. Sol. EtOH. Insol. H<sub>2</sub>O. Volatile in steam. Ox. → di-o-tolyl disulphide. H<sub>2</sub>SO<sub>4</sub> → blue col.

*S-Et*: C<sub>9</sub>H<sub>12</sub>S. MW, 152. Liq. B.p. 120°.

*S-Phenyl*: see Phenyl o-tolyl sulphide.

*S-o-Tolyl*: see Di-o-tolyl sulphide.

Hübner, Post, *Ann.*, 1873, 169, 30.

*m*-Thiocresol (*m*-Mercaptotoluene).

Liq. B.p. 195.4°, 126°/100 mm., 107.5°/50 mm. Sol. EtOH. Insol. H<sub>2</sub>O. Volatile in steam. D<sub>4</sub> 1.06251. Ox. → di-m-tolyl disulphide.

*S-Phenyl*: see Phenyl *m*-tolyl sulphide.

*S-m-Tolyl*: see Di-m-tolyl sulphide.

Hübner, Post, *Ann.*, 1873, 169, 51.

*p*-Thiocresol (*p*-Mercaptotoluene).

Plates from EtOH.Aq. or Et<sub>2</sub>O. M.p. 43–4°. B.p. 195°, 124.9°/100 mm., 71.4°/10.6 mm. Sol. EtOH, Et<sub>2</sub>O. H<sub>2</sub>SO<sub>4</sub> → blue col. Ox. → di-*p*-tolyl disulphide. Conc. H<sub>2</sub>SO<sub>4</sub> → 2 : 6-dimethylthianthrene. Produces eczema on skin.

*S-Me*: see Methyl *p*-tolyl sulphide.

*S-Et*: C<sub>9</sub>H<sub>12</sub>S. MW, 152. B.p. 220–1°, 105°/15 mm. D<sub>17</sub><sup>20</sup> 1.0016.

*S-Isopropyl*: C<sub>10</sub>H<sub>14</sub>S. MW, 166. Oil. B.p. 228°, 110°/14 mm.

*S-Phenyl*: see Phenyl *p*-tolyl sulphide.

*S-p-Tolyl*: see Di-*p*-tolyl sulphide.

Fischer, *Ber.*, 1915, 48, 96, 100.

Bourgeois, *Rec. trav. chim.*, 1899, 18, 437.

**Thiocresol-carboxylic Acid.**

See Mercaptotoluic Acid.

**Thio-*p*-cresotinic Acid.**See 4-Mercapto-*m*-toluic Acid.**Thiocyanic Acid**

CHNS

MW, 59

Gas. Forms colourless cryst. on cooling which rapidly melt at 5° to yellow liq. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O in all proportions. Dil. aq. sols. are stable.

*Me ester*: see Methyl thiocyanate.*Et ester*: see Ethyl thiocyanate.

*Propyl ester*: C<sub>4</sub>H<sub>9</sub>NS. MW, 101. Liq. with unpleasant odour. B.p. 163°.

*Isopropyl ester*: see Isopropyl thiocyanate.

*Butyl ester*: C<sub>5</sub>H<sub>9</sub>NS. MW, 115. B.p. 184.5–185.5°/743 mm. D<sub>4</sub><sup>25</sup> 0.9563.

*Isobutyl ester*: see Isobutyl thiocyanate.

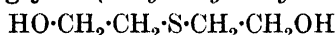
*Amyl ester*: C<sub>6</sub>H<sub>11</sub>NS. MW, 129. B.p. 90–1°/16 mm. D<sub>4</sub><sup>25</sup> 0.9412. n<sub>D</sub><sup>25</sup> 1.462.

*Isoamyl ester*: yellow oil. B.p. 197°.*Allyl ester*: see Allyl thiocyanate.

*Phenyl ester*: C<sub>7</sub>H<sub>5</sub>NS. MW, 135. Liq. B.p. 231°. D<sub>17</sub><sup>25</sup> 1.155.

*Benzyl ester*: see Benzyl thiocyanate.Allen, *J. Am. Chem. Soc.*, 1935, **57**, 198.Shriner, *Organic Syntheses*, 1931, **XI**, 92.Dienske, *Rec. trav. chim.*, 1927, **46**, 154.Kaufmann, Oehring, *Ber.*, 1926, **59**, 187.**Thiodiethylamine.**

See 2 : 2'-Diaminodiethyl sulphide.

**Thiodiglycol (Dihydroxydiethyl sulphide)**C<sub>4</sub>H<sub>10</sub>O<sub>2</sub>S

MW, 122

Syrup. B.p. 164–6°/20 mm. Misc. in all proportions with H<sub>2</sub>O. Combines with CaCl<sub>2</sub>.

*Diphenyl ether*: C<sub>16</sub>H<sub>18</sub>O<sub>2</sub>S. MW, 274. Needles from EtOH. M.p. 42°.

*Di-1-naphthyl ether*: C<sub>24</sub>H<sub>22</sub>O<sub>2</sub>S. MW, 374. Light brown cryst. M.p. 94.5°. Spar. sol. EtOH.

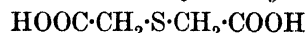
*Di-2-naphthyl ether*: cryst. M.p. 129°. Spar. sol. EtOH.

*Dibenzoyl*: cryst. from EtOH. M.p. 65°.*Di-p-nitrobenzoyl*: powder. M.p. 107.7°.*Di-p-aminobenzoyl*: powder. M.p. 184.5°.

Nenitzescu, Scărlătescu, *Ber.*, 1935, **68**, 588.

Faber, Miller, *Organic Syntheses*, 1932, **XII**, 68.

Helfrich, Reid, *J. Am. Chem. Soc.*, 1920, **42**, 1208.

**Thiodiglycollic Acid (Dimethyl sulphide dicarboxylic acid, dicarboxydimethyl sulphide)**C<sub>4</sub>H<sub>6</sub>O<sub>4</sub>S

MW, 150

Cryst. from AcOEt–C<sub>6</sub>H<sub>6</sub>. M.p. 129°. Sol. 2.37 parts H<sub>2</sub>O at 18°. Very sol. EtOH.  $k = 6.75 \times 10^{-4}$  at 25°. Ox. → dimethyl sulphone dicarboxylic acid. Red. → acetic acid. H<sub>2</sub>O<sub>2</sub> → thionyl diglycollic acid.

*Di-Me ester*: C<sub>6</sub>H<sub>10</sub>O<sub>4</sub>S. MW, 178. B.p. 135°/11 mm.

*Di-Et ester*: C<sub>8</sub>H<sub>14</sub>O<sub>4</sub>S. MW, 206. B.p. 267–8°.

*Monoamide*: C<sub>4</sub>H<sub>7</sub>O<sub>3</sub>NS. MW, 149. Prisms. M.p. 125°. Spar. sol. cold H<sub>2</sub>O. Sol. hot H<sub>2</sub>O. Heat → imide. *o-Toluidide*: needles from EtOH.Aq. M.p. 150–1°. *m-Toluidide*: cryst. from EtOH.Aq. M.p. 97–8°. *p-Toluidide*: plates from EtOH.Aq. M.p. 148–9°.

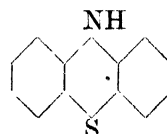
*Anhydride*: C<sub>4</sub>H<sub>4</sub>O<sub>3</sub>S. MW, 132. Needles from CHCl<sub>3</sub>. M.p. 102°. B.p. 158°/10 mm. Very sol. hot CHCl<sub>3</sub>. Spar. sol. hot Et<sub>2</sub>O.

*Nitrile*: C<sub>4</sub>H<sub>4</sub>N<sub>2</sub>S. MW, 112. Plates from MeOH. M.p. 45.5–46.5°.

*Monoanilide*: cryst. from H<sub>2</sub>O. M.p. 103°.*Dianilide*: needles from EtOH. M.p. 168°.

Beckurts, Frerichs, *J. prakt. Chem.*, 1906, **74**, 50.

Lovén, *Ber.*, 1894, **27**, 3059.

**Thiodiphenylamine (Phenthiazine)**C<sub>12</sub>H<sub>9</sub>NS

MW, 199

Yellow plates from EtOH. M.p. 182°. B.p. 371°, 290°/40 mm. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. cold EtOH, ligroin. Sublimes. Alc. FeCl<sub>3</sub> → green col. Heat with Zn → diphenylamine. Boil sol. with Cu → carbazole.

*N-Me*: C<sub>13</sub>H<sub>11</sub>NS. MW, 213. Exists in two forms. (α-) Long prisms from EtOH. M.p. 99.3°. B.p. 360–5°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. Et<sub>2</sub>O. Spar. sol. cold EtOH, AcOH. H<sub>2</sub>SO<sub>4</sub> → reddish-brown sol. (β-) Yellow needles from EtOH–C<sub>6</sub>H<sub>6</sub>. M.p. 78–9°. Sol. warm C<sub>6</sub>H<sub>6</sub>. Spar. sol. hot EtOH. Insol. H<sub>2</sub>O.

*N-Et*: C<sub>14</sub>H<sub>13</sub>NS. MW, 227. Prisms from EtOH. M.p. 102°.

*N-Phenyl*: C<sub>18</sub>H<sub>13</sub>NS. MW, 275. Cryst. from EtOH. M.p. 89–90°.

*N-Benzyl*: C<sub>19</sub>H<sub>15</sub>NS. MW, 289. Cryst. from EtOH. M.p. 130°.



**N-Acetyl**: prisms from EtOH. M.p. 197–197.5°.

Kehrmann, Dardel, *Ber.*, 1922, **55**, 2349.  
Knoevenagel, *J. prakt. Chem.*, 1914, **89**, 11.

Holzmann, *Ber.*, 1888, **21**, 2065.

Bernsthen, *Ann.*, 1885, **230**, 88.

### Thioethanolamine.

See 2-Mercaptoethylamine.

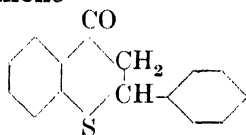
### Thioethyl Alcohol.

See Ethyl Mercaptan.

### Thioethylamine.

See 2 : 2'-Diaminodiethyl sulphide.

### Thioflavanone



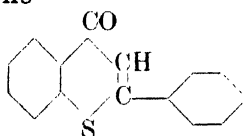
$C_{15}H_{12}OS$

MW, 240

Colourless needles from EtOH or pet. ether- $CS_2$ . M.p. 55–6°. Sol.  $Et_2O$ ,  $C_6H_6$ . Sol.  $H_2SO_4 \rightarrow$  red col. Insol. alkalis.

Arndt, *Ber.*, 1923, **56**, 1274.

### Thioflavone



$C_{15}H_{10}OS$

MW, 238

Needles from EtOH. M.p. 129–30° (125°). Alc. I  $\rightarrow$  blue col.  $H_2SO_4 \rightarrow$  yellow col. Forms salts readily hyd. by  $H_2O$ .

Arndt *et al.*, *Ber.*, 1925, **58**, 1620.

Ruhemann, *Ber.*, 1913, **46**, 2197.

### Thioformic Acid



$CH_2OS$

MW, 62

Unstable liq.

**Amide**: thioformamide.  $CH_3NS$ . MW, 61. Prisms from AcOEt or EtO–pet. ether. M.p. 28–9°. Very sol. EtOH,  $Me_2CO$ , AcOEt. Sol.  $Et_2O$ . Insol.  $CS_2$ , ligroin,  $C_6H_6$ , cold  $CHCl_3$ . Gradually decomp.

**Ethylamide**:  $C_3H_7NS$ . MW, 89. B.p. 125°/14 mm.

**Diethylamide**:  $C_5H_{11}NS$ . MW, 117. Oil. Cryst. on cooling. M.p. below 0°. B.p. 116–17°/14 mm. Spar. misc. with hot  $H_2O$ .

**Anilide**: thioformanilide. Needles from  $H_2O$ , plates from EtOH.Aq. M.p. 137.5° decomp.

**o-Toluidide**: needles from EtOH. M.p. 100–1°.

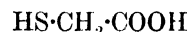
**p-Toluidide**: cryst. from EtOH. M.p. 173.5°.

Willstätter, Wirth, *Ber.*, 1909, **42**, 1911.

### Thioglucose.

See Glucothiose.

### Thioglycollic Acid (Mercaptoacetic acid)



$C_2H_4O_2S$

MW, 92

F.p. –16.5°. B.p. 123°/29 mm., 107–8°/16 mm.  $D^{20}_D$  1.3253. Oxidises in air.  $NH_3 + FeCl_3 \rightarrow$  dark red  $\rightarrow$  violet col.

**Et ester**:  $C_4H_8O_2S$ . MW, 120. B.p. 156–8°, 55°/17 mm.  $D^{15}_D$  1.0964. **Ag salt**: yellow needles from  $Me_2CO$ . M.p. 75–7°. **Hg salt**: needles from  $Et_2O$ . M.p. 56.5°. **Bi salt**: yellow cryst. from EtOH. M.p. 82.9°.

**Amide**:  $C_2H_5ONS$ . MW, 91. Needles. M.p. 52°. Very sol.  $H_2O$ . Spar. sol. EtOH. Oxidises in air.

**Anilide**: needles from  $H_2O$  or EtOH. M.p. 110.5–111°.

**o-Toluidide**: needles from EtOH.Aq. M.p. 84–5°.

**m-Toluidide**: needles from EtOH.Aq. M.p. 152–3°.

**p-Toluidide**: needles from EtOH. M.p. 125–6°.

**S-Et**:  $C_4H_8O_2S$ . MW, 120. Oil. F.p. –8.7°. B.p. 117–18°/11 mm.  $D^{20}_D$  1.1518. **Et ester**:  $C_6H_{12}O_2S$ . MW, 148. B.p. 187–9°.  $D^{20}_D$  1.0469. **Amide**:  $C_4H_9ONS$ . MW, 119. M.p. 44°.

**S-Propyl**:  $C_5H_{10}O_2S$ . MW, 134. Oil. **Amide**: m.p. 53°.

**S-Butyl**:  $C_6H_{12}O_2S$ . MW, 148. Yellow oil. B.p. 140–4°/10–15 mm. Insol.  $H_2O$ .

**S-Phenyl**:  $C_8H_8O_2S$ . MW, 168. Cryst. M.p. 60°. **Amide**: m.p. 103–4°.

**S-Benzyl**:  $C_9H_{10}O_2S$ . MW, 182. Cryst. M.p. 61–3°.

**S-Acetyl**: yellow oil. B.p. 158–9°/17 mm. **Chloride**: b.p. 93–5°/20 mm.

Holmberg, *J. prakt. Chem.*, 1934, **141**, 93.

Larsson, *Chem. Zentr.*, 1928, II, 234.

### Thioguaiacol.

See under Thiocatechol.

**Thiohydantoic Acid** (Thioureidoacetic acid, thiocarbaminylglycine, N-carboxymethylthiourea)



$C_3H_6O_2N_2S$

MW, 134

Needles or prisms from EtOH. M.p. 170–1° decomp. Very sol. hot H<sub>2</sub>O, EtOH. HgO → hydantoic acid.

*Et ester*: C<sub>5</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>S. MW, 162. Cryst. from H<sub>2</sub>O. M.p. about 65°. Very sol. H<sub>2</sub>O, EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. Et<sub>2</sub>O, pet. ether. 4-N-Acetyl: prisms. M.p. 104–5°. 4-N-Benzoyl: needles from EtOH. M.p. 128–9°.

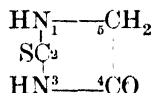
4-N-Acetyl: needles. M.p. 205° decomp.

4-N-Benzoyl: needles from EtOH, plates from H<sub>2</sub>O. M.p. 202°.

Wheeler, Nicolet, Johnson, *Am. Chem. J.*, 1911, **46**, 456.

Komatsu, *Chem. Zentr.*, 1911, II, 537.

## 2-Thiohydantoin (4-Keto-2-thiotetrahydro-*iminazole*)



C<sub>3</sub>H<sub>4</sub>ON<sub>2</sub>S

MW, 116

Yellow prisms from H<sub>2</sub>O. M.p. 228° decomp. Very sol. hot EtOH, Et<sub>2</sub>O, alkalis. Spar. sol. H<sub>2</sub>O. Gives cryst. K salt. Hot aq. Ba(OH)<sub>2</sub> → thiohydantoic acid.

3-N-Me: C<sub>4</sub>H<sub>6</sub>ON<sub>2</sub>S. MW, 130. Needles from CHCl<sub>3</sub>-ligroin. M.p. 161°. Very sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. CHCl<sub>3</sub>. Insol. ligroin.

1:3-N-Di-Me: C<sub>5</sub>H<sub>8</sub>ON<sub>2</sub>S. MW, 144. Cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 94.5°. Very sol. hot H<sub>2</sub>O. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

3-N-Allyl: C<sub>6</sub>H<sub>8</sub>ON<sub>2</sub>S. MW, 156. Cryst. from ligroin. M.p. 108°. Sol. hot H<sub>2</sub>O, EtOH, AcOH. Spar. sol. CHCl<sub>3</sub>, ligroin, C<sub>6</sub>H<sub>6</sub>.

3-N-Phenyl: C<sub>9</sub>H<sub>8</sub>ON<sub>2</sub>S. MW, 192. Yellow plates from EtOH. M.p. 240–2° decomp.

1:3-N-Diphenyl: C<sub>15</sub>H<sub>12</sub>ON<sub>2</sub>S. MW, 268. Yellow prisms from AcOH, needles from EtOH. M.p. 212°. Sol. hot AcOH. Spar. sol. Et<sub>2</sub>O, EtOH, ligroin, C<sub>6</sub>H<sub>6</sub>.

3-N-Benzyl: C<sub>10</sub>H<sub>10</sub>ON<sub>2</sub>S. MW, 206. Cryst. from EtOH. M.p. 128°.

3-N-o-Tolyl: yellow plates from EtOH. M.p. 149–50°.

3-N-p-Tolyl: yellow cryst. from EtOH. M.p. 228°.

1-N-Acetyl: plates from EtOH. M.p. 175–6°.

1-N-Benzoyl: prisms from EtOH. M.p. 165°.

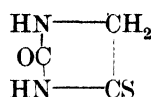
Johnson, Hill, Bailey, *J. Am. Chem. Soc.*, 1915, **37**, 2406.

Johnson, *Am. Chem. J.*, 1913, **49**, 68.

Marckwald, Neumark, Stelzner, *Ber.*, 1891, **24**, 3285.

Dict. of Org. Comp.—III.

## 4-Thiohydantoin



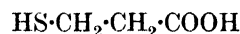
C<sub>3</sub>H<sub>4</sub>ON<sub>2</sub>S

MW, 116

Needles from hot H<sub>2</sub>O. Decomp. above 200°. Sol. NaOH → red sol.

Johnson, Chernoff, *J. Am. Chem. Soc.*, 1912, **34**, 1208.

## Thiohydracrylic Acid (2-Mercaptopropionic acid)



C<sub>3</sub>H<sub>6</sub>O<sub>2</sub>S

MW, 106

Cryst. M.p. 16–8°. B.p. 110.5–111.5°. D<sub>20</sub><sup>20</sup> 1.218. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Ox. → di-thiohydracrylic acid. FeCl<sub>3</sub> → blue col.

Billmann, *Ann.*, 1906, **348**, 126.

Lovén, *J. prakt. Chem.*, 1884, **29**, 376.

## Thiohydroquinone (p-Mercaptophenol, p-hydroxythiophenol)



C<sub>6</sub>H<sub>6</sub>OS

MW, 126

Cryst. with strong odour. M.p. 29–30°. B.p. 166–8°/45 mm., 133–7°/11 mm. Sol. H<sub>2</sub>O.

O-Me ether: p-mercaptoanisole, p-methoxythiophenol. C<sub>7</sub>H<sub>8</sub>OS. MW, 140. B.p. 227°, 89–90°/5 mm. D<sub>4</sub><sup>25</sup> 1.1313. n<sub>D</sub><sup>25</sup> 1.5801. Volatile in steam.

S-Me: p-hydroxythioanisole. Plates from pet. ether. M.p. 84–5°. Me ether: p-methoxythioanisole. C<sub>8</sub>H<sub>10</sub>OS. MW, 154. M.p. 25–6°. B.p. 99–100°/4 mm. D<sub>4</sub><sup>25</sup> 1.1069. n<sub>D</sub><sup>25</sup> 1.5764. Et ether: p-ethoxythioanisole. C<sub>9</sub>H<sub>12</sub>OS. MW, 168. M.p. 19–20°. B.p. 98–100°/5 mm. D<sub>4</sub><sup>25</sup> 1.0693. n<sub>D</sub><sup>25</sup> 1.5618. Acetyl: needles from pet. ether. M.p. 43–4°.

O-Et ether: p-mercaptophenetole. C<sub>8</sub>H<sub>10</sub>OS. MW, 154. B.p. 232–5°. Volatile in steam.

S-Et: p-hydroxythiophenetole. Cryst. from pet. ether. M.p. 39–41°. Me ether: p-methoxythiophenetole. C<sub>9</sub>H<sub>12</sub>OS. MW, 168. B.p. 103°/5 mm. D<sub>4</sub><sup>25</sup> 1.0674. n<sub>D</sub><sup>25</sup> 1.5600. Et ether: p-ethoxythiophenetole. C<sub>10</sub>H<sub>14</sub>OS. MW, 182. B.p. 110–12°/6 mm.

S-Propyl: propyl p-hydroxyphenyl sulphide. C<sub>9</sub>H<sub>12</sub>OS. MW, 168. Cryst. from pet. ether. M.p. 33–33.5°. Me ether: propyl p-methoxyphenyl sulphide. C<sub>10</sub>H<sub>14</sub>OS. MW, 182. B.p. 110–11°/5 mm. D<sub>4</sub><sup>25</sup> 1.0424. n<sub>D</sub><sup>25</sup> 1.5545.

**S-Butyl**: butyl *p*-hydroxyphenyl sulphide.  $C_{10}H_{14}OS$ . MW, 182. Cryst. from pet. ether. M.p. 49–50°. **Me ether**: butyl *p*-methoxyphenyl sulphide.  $C_{11}H_{16}OS$ . MW, 196. B.p. 120°/5 mm.  $D_4^{25}$  1.0303.  $n_D^{25}$  1.5445.

**S-n-Amyl**: *n*-amyl *p*-hydroxyphenyl sulphide.  $C_{11}H_{16}OS$ . MW, 196. Cryst. from pet. ether. M.p. 55–6°. **Me ether**: *n*-amyl *p*-methoxyphenyl sulphide.  $C_{12}H_{18}OS$ . MW, 210. B.p. 127°/5 mm.  $D_4^{25}$  1.0149.  $n_D^{25}$  1.5380.

**S-n-Hexyl**: *n*-hexyl *p*-hydroxyphenyl sulphide.  $C_{12}H_{18}OS$ . MW, 210. Cryst. from pet. ether. M.p. 58–9°. **Me ether**: *n*-hexyl *p*-methoxyphenyl sulphide.  $C_{13}H_{20}OS$ . MW, 224. B.p. 142°/5 mm.  $D_4^{25}$  0.9975.  $n_D^{25}$  1.5315.

**S-Phenyl**: *p*-hydroxydiphenyl sulphide.  $C_{12}H_{10}OS$ . MW, 202. Prisms from pet. ether. M.p. 50–1°. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. **Me ether**: *p*-methoxydiphenyl sulphide.  $C_{13}H_{12}OS$ . MW, 216. Plates from EtOH.Aq. M.p. 88°. **p-Nitrobenzoyl**: yellow prisms from EtOH. M.p. 74–5°.

**S-p-Tolyl**: *p*-tolyl *p*-hydroxyphenyl sulphide.  $C_{13}H_{12}OS$ . MW, 216. Plates from pet. ether. M.p. 67–8°. Spar. sol. H<sub>2</sub>O. **Me ether**: *p*-tolyl *p*-methoxyphenyl sulphide.  $C_{14}H_{14}OS$ . MW, 230. Plates from EtOH. M.p. 45–6°. B.p. 181–2°/4 mm.

Miller, Read, *J. Am. Chem. Soc.*, 1933, **55**, 1224.

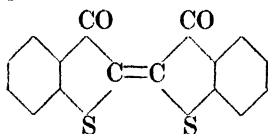
Suter, Hansen, *J. Am. Chem. Soc.*, 1932, **54**, 4101.

Zincke, Ebel, *Ber.*, 1914, **47**, 1104.

Gattermann, *Ber.*, 1899, **32**, 1148.

Leuckart, *J. prakt. Chem.*, 1890, **41**, 192.

### Thioindigo



$C_{16}H_8O_2S_2$

MW, 296

Brownish-red needles with bronze reflex from xylene, red cryst. from C<sub>6</sub>H<sub>6</sub>. Does not melt below 280°. Very sol. hot PhNO<sub>2</sub>. Spar. sol. hot EtOH, CHCl<sub>3</sub>, CS<sub>2</sub>. Sublimes. Red sol. in xylene shows reddish-yellow fluorescence. Sol. H<sub>2</sub>SO<sub>4</sub> → bluish-green or blue col. Fairly stable to ox. agents. HNO<sub>3</sub> → thioindigo-S-oxide. Red. → Thioindigo White.

**S-Oxide**:  $C_{16}H_8O_3S_2$ . MW, 312. Scarlet cryst. from CHCl<sub>3</sub> or xylene. M.p. about 325°

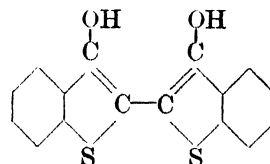
decomp. Sol. CHCl<sub>3</sub>, PhNO<sub>2</sub>, Py. Spar. sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. H<sub>2</sub>SO<sub>4</sub> → violet sol.

Jezierski, *Chem. Abstracts*, 1935, **29**, 2161.

Kalle, D.R.P., 194,254, (*Chem. Zentr.*, 1908, I, 1116).

Friedländer, *Ann.*, 1907, **351**, 411.

### Thioindigo White (Leucothioindigo)



$C_{16}H_{10}O_2S_2$

MW, 298

White ppt. Turns red in air. Sol. most org. solvents. Very sol. dil. NaOH → sol. which reddens in air. Insol. H<sub>2</sub>O.

**Diacetyl**: colourless needles from Ac<sub>2</sub>O or xylene. M.p. 248° decomp. (240°).

**Monobenzoyl**: needles from xylene. M.p. 225° decomp.

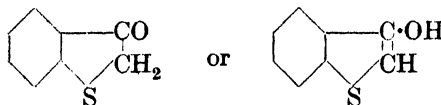
**Dibenzoyl**: cryst. from xylene. M.p. 227°.

Posner, Wallis, *Ber.*, 1924, **57**, 1673.

Tschilikin, *Chem. Zentr.*, 1916, I, 942.

Béchamp, *Compt. rend.*, 1909, **148**, 1678.

### Thioindoxyl (3-Hydroxythionaphthene)



$C_8H_6OS$

MW, 150

Needles from H<sub>2</sub>O. M.p. 71°. Very sol. most org. solvents. Spar. sol. cold H<sub>2</sub>O. Sol. alkalis in which it is readily oxidised. Turns red in air.

**Me ether**:  $C_9H_8OS$ . MW, 164. Oil. B.p. 260–1°. **Picrate**: brownish-red needles from EtOH. M.p. 112°.

**Et ether**:  $C_{10}H_{10}OS$ . MW, 178. Oil. B.p. 154°/19 mm.  $D_4^{25}$  1.1591.

**Acetyl**: yellow oil. B.p. 165°/18 mm.

**Semicarbazone**: needles from EtOH. M.p. 224–5°.

**p-Nitrophenylhydrazones**: exists in two forms.

(i) Reddish-brown powder. M.p. 185–90°.

(ii) Reddish-brown powder. M.p. 251–61°.

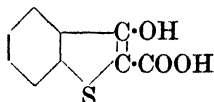
McClelland, D'Silva, *J. Chem. Soc.*, 1931, 2974.

Auwers, Thies, *Ber.*, 1920, **53**, 2291.

Friedländer, *Ann.*, 1907, **351**, 408.

Auwers, *Ann.*, 1912, **393**, 379.

**Thioindoxyllic Acid** (3-Hydroxythionaphthene-2-carboxylic acid, thioindoxyl-2-carboxylic acid).



$C_9H_6O_3S$

MW, 194

Free acid is very unstable and loses  $CO_2$  readily. Very sol. EtOH, AcOH. Sol.  $H_2O$ .

*Me ether*:  $C_{10}H_8O_3S$ . MW, 208. Prisms from MeOH. M.p.  $173^\circ$ . Sol. MeOH, EtOH. Spar. sol.  $C_6H_6$ , ligroin.

*Et ether*:  $C_{11}H_{10}O_3S$ . MW, 222. Prisms from MeOH. M.p.  $158^\circ$ . Very sol. EtOH. Sol.  $C_6H_6$ . Spar. sol. ligroin.

*Me ester*:  $C_{10}H_8O_3S$ . MW, 208. Cryst. from EtOH.Aq. M.p.  $107-8^\circ$  ( $104^\circ$ ).

*Et ester*:  $C_{11}H_{10}O_3S$ . MW, 222. Cryst. from ligroin. M.p.  $73-4^\circ$ .

*Methylamide*: red needles from MeOH. M.p.  $122-3^\circ$ .

*Ethylamide*: needles from EtOH.Aq. M.p.  $135^\circ$ .

*Benzylamide*: needles from EtOH. M.p.  $134^\circ$ .

*Anilide*: needles from EtOH. M.p.  $231^\circ$ .

*Acetyl*: needles from EtOH. M.p.  $180^\circ$ .

Auwers, *Ann.*, 1912, **393**, 372.

Friedländer, *Ann.*, 1907, **351**, 405.

**Thioisoamyl Alcohol.**

See Isoamyl Mercaptan.

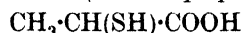
**Thioisobutyl Alcohol.**

See Isobutyl Mercaptan.

**Thioisopropyl Alcohol.**

See Isopropyl Mercaptan.

**Thiolactic Acid** (1-Mercaptopropionic acid)



$C_3H_6O_2S$

MW, 106

*d.*

B.p.  $95-100^\circ/16$  mm.  $[\alpha]_D^{20} + 38.32^\circ$ .

*l.*

Oil.  $D^{19.2} 1.193$ .  $[\alpha]_D^{15} - 45.47^\circ$ .

*dl.*

Oil. B.p.  $98.5-99^\circ/14$  mm. Sol.  $H_2O$ , EtOH,  $Et_2O$ .  $FeCl_3 \rightarrow$  indigo-blue col.

*Et ester*:  $C_5H_{10}O_2S$ . MW, 134. Liq. Spar. sol.  $H_2O$ .

*S-Benzyl*: prisms. M.p.  $73-4^\circ$ .

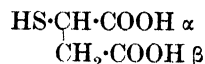
Lovén, *J. prakt. Chem.*, 1884, **29**, 368; 1908, **78**, 65.

Billmann, *Ann.*, 1906, **348**, 124.

Levene, Mikeska, *J. Biol. Chem.*, 1924, **60**, 1.

Larsson, *Chem. Abstracts*, 1928, **22**, 4470.

**Thiomalic Acid** (Mercaptosuccinic acid)



$C_4H_6O_4S$

MW, 150

*d.*

M.p.  $152-3^\circ$  ( $138^\circ$ ).  $[\alpha]_D^{17} + 64.4^\circ$  in EtOH.

*$\beta$ -Monoamide*:  $C_4H_7O_3NS$ . MW, 149. M.p.  $125^\circ$ .  $[\alpha]_D^{18} + 82.5^\circ$  in  $Me_2CO$ .

*l.*

M.p.  $152-3^\circ$   $[\alpha]_D^{17} - 64.8^\circ$  in EtOH.

*$\beta$ -Monoamide*: m.p.  $125^\circ$ .  $[\alpha]_D^{18} - 82.9^\circ$  in  $Me_2CO$ .

*dl.*

Cryst. M.p.  $149-50^\circ$ . Sol.  $H_2O$ , EtOH,  $Me_2CO$ . Mod. sol.  $Et_2O$ . Very spar. sol.  $C_6H_6$ .  $k = 5.23 \times 10^{-4}$  at  $25^\circ$ .  $FeCl_3 \rightarrow$  blue col.

*Di-Et ester*:  $C_8H_{14}O_4S$ . MW, 206. Oil. B.p. about  $246^\circ$  part. decomp.

*$\beta$ -Monoamide*: cryst. from EtOH. M.p.  $103^\circ$ .

*S-Benzyl*: needles from EtOH.Aq. M.p.  $181^\circ$ .

Holmberg, Lenander, *Chem. Zentr.*, 1918, I, 1146.

Billmann, *Ann.*, 1905, **339**, 371.

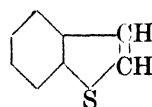
Levene, Mikeska, *J. Biol. Chem.*, 1924, **60**, 685.

Holmberg, *Chem. Zentr.*, 1916, I, 968.

**Thiomorpholine.**

See 1:4-Thiazan.

**Thionaphthene** (2:3-Benzthiophene, thio-coumarone)



$C_8H_6S$

MW, 134

Present in lignite tar. Leaflets with odour resembling naphthalene. M.p.  $32^\circ$ . B.p.  $221-2^\circ$ . Sol. common org. solvents. Volatile in steam. Sol. conc.  $H_2SO_4$  with cherry-red col. disappearing on heating.  $H_2O_2 \rightarrow$  S-oxide.

*Picrate*: yellow needles from EtOH. M.p.  $149^\circ$ .

*S-Oxide*:  $C_8H_6O_2S$ . MW, 166. M.p.  $142^\circ$ .

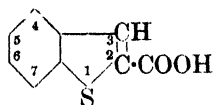
Gattermann, Lockhart, *Ber.*, 1893, **26**, 2808.

Bezdrík, Friedländer, Koeniger, *Ber.*, 1908, **41**, 231, 236.

Chmielewsky, Friedländer, *Ber.*, 1913, **46**, 1907.

Weissgerber, Kruber, *Ber.*, 1920, **53**, 1551.

## Thionaphthene-2-carboxylic Acid

 $C_9H_6O_2S$ 

MW, 178

Needles from  $H_2O$ . M.p.  $236^\circ$  ( $114^\circ$ ). Very sol.  $Et_2O$ . Spar. sol.  $C_6H_6$ , cold  $H_2O$ . Hot alkalis  $\rightarrow$  thionaphthene.

*Me ester*:  $C_{10}H_8O_2S$ . MW, 192. Prisms from  $EtOH$ . M.p.  $72-3^\circ$ . B.p.  $176-80^\circ/13$  mm.

*Et ester*:  $C_{11}H_{10}O_2S$ . MW, 206. Cryst. from  $EtOH$ . M.p.  $36-7^\circ$ .

*Chloride*:  $C_9H_5OClS$ . MW, 196.5. Leaflets from ligroin. M.p.  $88-9^\circ$ . B.p.  $173-5^\circ/19$  mm.

*Amide*:  $C_9H_7ONS$ . MW, 177. Needles from  $H_2O$ . M.p.  $177^\circ$ .

*Hydrazide*: leaflets from  $EtOH$ . M.p.  $184-5^\circ$ .

*Azide*: needles from  $EtOH$ . M.p.  $108^\circ$  decomp.

Ges. für Teerverwertung, D.R.P., 341,837, (Chem. Zentr., 1921, IV, 1225).

Weissgerber, Kruber, Ber., 1920, 53, 1561.

Friedländer, Lenk, Ber., 1912, 45, 2087.

## Thionaphthene-3-carboxylic Acid.

Cryst. from  $EtOH.Aq$ . M.p.  $174-5^\circ$ . Sol. common org. solvents. Prac. insol.  $H_2O$ . Volatile in steam.

*Me ester*: oil. B.p.  $285-7^\circ/750$  mm.,  $165-6^\circ/17$  mm.

*Et ester*: oil. B.p.  $304-6^\circ/750$  mm.,  $172-3^\circ/17$  mm.

*Chloride*: m.p. about  $50^\circ$ . B.p.  $296-8^\circ/758$  mm.

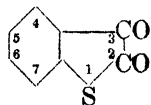
*Amide*: needles from  $EtOH$ . M.p.  $197-8^\circ$ .

*Anilide*: needles from  $C_6H_6$ . M.p.  $172-3^\circ$ .

Komppa, Weekman, J. prakt. Chem., 1933, 138, 116.

Komppa, J. prakt. Chem., 1929, 122, 331.

**Thionaphthenequinone** (2:3-Diketodihydrothionaphthene, 2:3-diketothiocoumaran, thio-coumarandione)

 $C_8H_4O_2S$ 

MW, 164

Yellow prisms from  $EtOH$ . M.p.  $121^\circ$ . B.p. about  $247^\circ$ . Sol.  $EtOH$ ,  $Me_2CO$ ,  $AcOH$ ,  $C_6H_6$ . Spar. sol. cold ligroin. Mod. volatile in steam. Sol. dil.  $NaOH$  with orange-yellow col.  $\rightarrow$  2-mercaptobenzoylformic acid.

*2-Oxime*: yellowish-red cryst. from  $EtOH$ . M.p.  $170-1^\circ$ . *O-Me ether*: reddish-yellow

needles from  $EtOH$ . M.p.  $125^\circ$ . *Acetyl*: yellow needles from xylene. M.p.  $174^\circ$  ( $168^\circ$ ). *Benzoyl*: yellow plates from  $C_6H_6$ . M.p.  $170^\circ$ . *Benzene-sulphonyl*: yellow needles from xylene. M.p.  $231^\circ$ . *Phenylhydrazone*: yellow needles from  $EtOH$ . M.p.  $172^\circ$ .

*3-Oxime*: yellow needles from  $EtOH.Aq$ . M.p.  $186^\circ$ .

*3-Phenylhydrazone*: 3-benzeneazo-2-hydroxythionaphthene. Orange-red cryst. from  $EtOH$ . M.p.  $165-6^\circ$ .

*Di-phenylhydrazone*: orange-red needles from  $C_6H_6$ . M.p.  $199-200^\circ$ .

*3-p-Nitrophenylhydrazone*: red cryst. from xylene. M.p.  $271-2^\circ$ .

*2-Anil*: yellowish-red needles from  $EtOH$ . M.p.  $151-2^\circ$ .

*2-p-Tolil*: brownish-red needles from  $EtOH$ . M.p.  $159^\circ$ .

Kalle, D.R.P., 241,623, (Chem. Zentr., 1912, I, 174).

Bezdrík, Friedländer, Koeniger, Ber., 1908, 41, 235.

**1-Thionaphthol** (1-Naphthyl mercaptan, 1-mercaptanaphthalene,  $\alpha$ -thionaphthol)

 $C_{10}H_8S$ 

MW, 160

B.p.  $208.5^\circ/200$  mm.,  $187.2^\circ/50$  mm.,  $161^\circ/20$  mm.,  $144.8^\circ/10.3$  mm.  $D_4^{25}$  1.1549. Sol.  $EtOH$ ,  $Et_2O$ . Spar. sol. aq. alkalis. Volatile in steam.

*Me ether*: see Methyl 1-naphthyl sulphide.

*Et ether*: ethyl 1-naphthyl sulphide.  $C_{12}H_{12}S$ . MW, 188. B.p.  $175-6^\circ/25$  mm.,  $167-167.5^\circ/15$  mm.  $D_4^{25}$  1.1198,  $D_4^{20}$  1.0797.

*Phenyl ether*: see Phenyl 1-naphthyl sulphide.

*o-Tolyl ether*: o-tolyl 1-naphthyl sulphide.  $C_{17}H_{14}S$ . MW, 250. Oil. B.p.  $227.5^\circ/11$  mm.  $D_4^{25}$  1.1504.

*m-Tolyl ether*: m-tolyl 1-naphthyl sulphide. Oil. B.p.  $229^\circ/11$  mm.  $D_4^{25}$  1.1445.

*p-Tolyl ether*: p-tolyl 1-naphthyl sulphide. Cryst. from  $EtOH$ . M.p.  $40.5^\circ$ . B.p.  $232.5^\circ/11$  mm.

*Naphthyl ether*: see Dinaphthyl sulphide.

*Benzyl ether*: benzyl 1-naphthyl sulphide. Leaflets from  $EtOH$ . M.p.  $78-80^\circ$ .

*Acetyl*: pale yellow liq. B.p.  $200-3^\circ/25$  mm.,  $188^\circ/15$  mm.  $D_4^{25}$  1.1519.

Bourgeois, Rec. trav. chim., 1899, 18, 441; Ber., 1895, 28, 2328.

Fichter, Tamm, Ber., 1910, 43, 3033.

Leuckart, J. prakt. Chem., 1890, 41, 216.

**2-Thionaphthol** (2-Naphthyl mercaptan, 2-mercaptanaphthalene,  $\beta$ -thionaphthol).

Cryst. from EtOH. M.p.  $81^{\circ}$ . B.p.  $286^{\circ}$ ,  $210.5^{\circ}/100$  mm.,  $189^{\circ}/50$  mm.,  $162.7^{\circ}/20$  mm.,  $153.5^{\circ}/15$  mm.,  $146.3^{\circ}/10.3$  mm. Very sol. EtOH, Et<sub>2</sub>O, pet. ether. Spar. sol. H<sub>2</sub>O. Spar. volatile in steam.

*Me ether*: see Methyl 2-naphthyl sulphide.

*Et ether*: ethyl 2-naphthyl sulphide. M.p.  $16^{\circ}$ . B.p.  $170.5^{\circ}/15$  mm.

*Phenyl ether*: see Phenyl 2-naphthyl sulphide.

*o-Tolyl ether*: *o*-tolyl 2-naphthyl sulphide. Oil. B.p.  $229.5^{\circ}/11$  mm. D<sub>4</sub><sup>20</sup> 1.1420.

*m-Tolyl ether*: *m*-tolyl 2-naphthyl sulphide. Needles from EtOH.Aq. M.p.  $60^{\circ}$ . B.p.  $235^{\circ}/11$  mm.

*p-Tolyl ether*: *p*-tolyl 2-naphthyl sulphide. Leaflets from EtOH.Aq. M.p.  $68^{\circ}$ . B.p.  $237^{\circ}/11$  mm.

*Naphthyl ether*: see Dinaphthyl sulphide.

*Acetyl*: m.p.  $53.5^{\circ}$ . B.p.  $191^{\circ}/15$  mm.

Zincke, Eismayer, *Ber.*, 1918, **51**, 755.

Leuckart, *J. prakt. Chem.*, 1890, **41**, 219.

Fichter, Tamm, *Ber.*, 1910, **43**, 3034.

**Thioneine**.

See Ergothioneine.

**Thionessal**.

See Tetraphenylthiophene.

**Thionylaniline**



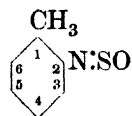
$\text{C}_6\text{H}_5\text{ONS}$

MW, 139

Yellow liq. with acrid aromatic odour. B.p.  $200^{\circ}$ . D<sub>4</sub><sup>20</sup> 1.2360. Sol. EtOH. Decomp. by H<sub>2</sub>O, dil. acids, alkalis,  $\rightarrow$  aniline + SO<sub>2</sub>. Cl in pet. ether  $\rightarrow$  2 : 4 : 6-trichloroaniline hydrochloride.

Michaelis, *Ber.*, 1891, **24**, 746.

**Thionyl-*o*-toluidine**



$\text{C}_7\text{H}_7\text{ONS}$

MW, 153

Liq. B.p.  $184^{\circ}/100$  mm.

Michaelis, *Ann.*, 1893, **274**, 226.

**Thionyl-*m*-toluidine**.

Yellow oil. B.p.  $220^{\circ}$ . Volatile in steam.

See previous reference.

**Thionyl-*p*-toluidine**.

Yellow cryst. M.p.  $9^{\circ}$ . B.p.  $224^{\circ}$ . D<sub>4</sub><sup>20</sup> 1.1685.

See previous reference.

**Thio-oxalic Acid**



$\text{C}_2\text{H}_2\text{O}_3\text{S}$

MW, 106

Free acid does not exist.

*Di-Me ester*:  $\text{C}_4\text{H}_6\text{O}_3\text{S}$ . MW, 134. B.p.  $50-3^{\circ}/21$  mm.

*Di-Et ester*:  $\text{C}_6\text{H}_{10}\text{O}_3\text{S}$ . MW, 162. B.p.  $217^{\circ}$ . D<sub>4</sub><sup>20</sup> 1.1446. Hyd.  $\rightarrow$  oxalic acid.

*S-p-Tolyl ester*:  $\text{C}_9\text{H}_8\text{O}_3\text{S}$ . MW, 196. Plates or needles from pet. ether. M.p.  $100^{\circ}$  decomp. Very sol. Et<sub>2</sub>O, EtOH. Spar. sol. pet. ether. Sol. H<sub>2</sub>O with decomp. *Anilide*: needles from EtOH. M.p.  $137^{\circ}$ .

*Amide*: see Thio-oxamic Acid.

*Diamide*: see under Thio-oxamic Acid.

*Nitrile*:  $\text{C}_2\text{HONS}$ . MW, 87. *Anilide*: orange-yellow needles. M.p.  $82^{\circ}$ . *o-Toluidide*: m.p.  $64^{\circ}$ .

*Anilide*: yellow needles from H<sub>2</sub>O, yellow plates from EtOH.Aq. M.p.  $101-2^{\circ}$ . *Amide*: oxanilic acid thioamide. Yellow prisms from EtOH. M.p.  $176^{\circ}$ .

*Dianilide*: thio-oxanilide. Yellow needles from AcOH or EtOH. M.p.  $144-5^{\circ}$ .

*Di-o-toluidide*: yellow needles and plates from EtOH. M.p.  $126^{\circ}$ .

*Di-m-toluidide*: yellow needles and plates from EtOH. M.p.  $88-9^{\circ}$ .

*Di-p-toluidide*: yellow needles from EtOH. M.p.  $153-4^{\circ}$ .

Reissert, *Ber.*, 1904, **37**, 3720.

Morley, Saint, *J. Chem. Soc.*, 1883, **43**, 400.

**Thio-oxamic Acid**



$\text{C}_2\text{H}_3\text{O}_2\text{NS}$

MW, 105

Exists only as salts and esters.

*Me ester*:  $\text{C}_3\text{H}_5\text{O}_2\text{NS}$ . MW, 119. Yellow needles. M.p.  $86^{\circ}$ . Very sol. EtOH, Et<sub>2</sub>O. Sol. H<sub>2</sub>O.

*Et ester*:  $\text{C}_4\text{H}_7\text{O}_2\text{NS}$ . MW, 133. Yellow prisms. M.p.  $63^{\circ}$ . Very sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

*Isobutyl ester*:  $\text{C}_6\text{H}_{11}\text{O}_2\text{NS}$ . MW, 161. Yellow needles or prisms from EtOH. M.p.  $58^{\circ}$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

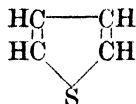
*Amide*: thio-oxamide.  $\text{C}_2\text{H}_4\text{ON}_2\text{S}$ . MW, 104. Yellow needles from EtOH. Spar. sol. cold H<sub>2</sub>O, cold EtOH.

*Nitrile*: cyanthioformamide.  $\text{C}_2\text{H}_2\text{N}_2\text{S}$ . Yellow needles. M.p.  $87-90^{\circ}$  decomp. Sol. H<sub>2</sub>O. Very unstable.

*Anilide*: thio-oxanilic acid amide. Yellow needles from EtOH. M.p. 169–70°.

Weddige, *J. prakt. Chem.*, 1874, 10, 200; 9, 133.

### Thiophene



$C_4H_4S$

MW, 84

F.p. –37.1°. M.p. 29.8°. B.p. 84°.  $D_4^{20}$  1.0617.  $n_D^{20}$  1.5246. Heat of comb.  $C_p$  670.9 Cal.,  $C_v$  669.5 Cal., (vapour)  $C_p$  610.6 Cal. Red hot tube  $\rightarrow$  2:2'-dithienyl + 3:3'-dithienyl. Sol.  $H_2SO_4 \rightarrow$  red  $\rightarrow$  deep brown col.  $H_2SO_4$  + nitrite  $\rightarrow$  blue col. Isatin +  $H_2SO_4 \rightarrow$  indophenin. Thalline in pet. ether +  $HNO_3 \rightarrow$  intense violet col.  $\rightarrow$  red  $\rightarrow$  yellow: disappears with  $H_2O$ .

Jurjew, *Ber.*, 1936, 69, 440, 1002.

Phillips, *Organic Syntheses*, 1932, XII, 72.

I.G., E.P., 305,603.

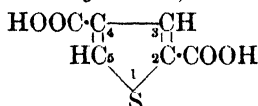
### Thiophene-acetic Acid.

See Thienylacetic Acid.

### Thiophene-carboxylic Acid.

See Thiophenic Acid.

**Thiophene-2:4-dicarboxylic Acid** (*Thiophene- $\alpha$ :  $\beta'$ -dicarboxylic acid*)



$C_6H_4O_4S$

MW, 172

Decomp. at 280° with part. sublimation. Very sol. hot  $H_2O$ , spar. sol. cold.

*Di-Me ester*:  $C_8H_8O_4S$ . MW, 200. Plates from EtOH.Aq. M.p. 120–1°.

*Di-Et ester*:  $C_{10}H_{12}O_4S$ . MW, 228. M.p. 35–6°.

Zelinsky, *Ber.*, 1887, 20, 2021.

**Thiophene-2:5-dicarboxylic Acid** (*Thiophene- $\alpha$ :  $\alpha'$ -dicarboxylic acid*).

Cryst. powder. Does not melt below 350°. Sol.  $Et_2O$ . Spar. sol.  $H_2O$ . Sublimes.

*Di-Me ester*: needles from  $Et_2O$ , prisms from EtOH. M.p. 151° (145°).

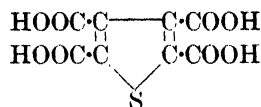
*Di-Et ester*: needles from EtOH. M.p. 51.5°. Sol. EtOH.

*Dinitrile*:  $C_6H_2N_2S$ . MW, 134. Cryst. from  $Et_2O$ . M.p. 92–92.5°.

Hinsberg, *Ber.*, 1912, 45, 2414.

Jaekel, *Ber.*, 1886, 19, 190.

### Thiophene-tetracarboxylic Acid



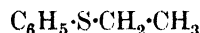
$C_8H_4O_8S$

MW, 260

*Tetra-Me ester*:  $C_{12}H_{12}O_8S$ . MW, 316. Prisms from EtOH. M.p. 126–8°. Very sol. hot EtOH, AeOEt. Spar. sol. hot  $H_2O$ .

Michael, *Ber.*, 1895, 28, 1635.

### Thiophenetole (*Ethyl phenyl sulphide*)



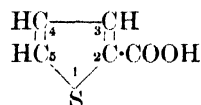
$C_8H_{10}S$

MW, 138

Liq. with unpleasant odour. B.p. 204°.  $D_4^{20}$  1.024.  $n_D^{20}$  1.5662.  $KMnO_4 \rightarrow$  ethyl phenyl sulphone.

Otto, *Ber.*, 1880, 13, 1275.

**$\alpha$ -Thiophenic Acid** (*Thiophene-2-carboxylic acid*)



$C_5H_4O_2S$

MW, 128

Needles from  $H_2O$ . M.p. 126–7°. Very sol. hot  $H_2O$ , EtOH,  $Et_2O$ . Sol.  $CHCl_3$ . Spar. sol. pet. ether. Sol. 140 parts  $H_2O$  at 25°. Has irritating odour.  $k = 3.2 \times 10^{-4}$  at 25°. Heat of comb.  $C_v$  645.4 Cal.,  $C_p$  646.3 Cal. Red.  $\rightarrow$  tetrahydrothiophene-2-carboxylic acid. Dist. Ca salt  $\rightarrow$  2:2'-dithienyl ketone. Isatin +  $H_2SO_4 \rightarrow$  blue sol.

*Et ester*:  $C_7H_8O_2S$ . MW, 156. B.p. 218°, 115°/25 mm., 96°/18 mm.  $D_4^{18.5}$  1.1623.

*Chloride*:  $C_5H_3OClS$ . MW, 146.5. B.p. 190°.

*Amide*:  $C_5H_7ONS$ . MW, 127. Cryst. powder from EtOH. M.p. 180° (174°). Spar. sol.  $Et_2O$ .

*Anhydride*:  $C_{10}H_6O_3S_2$ . MW, 238. Cryst. M.p. 62°. B.p. 218–20°/15 mm.

*Nitrile*: 2-oyanthiophene.  $C_6H_3NS$ . MW, 109. Oil. B.p. 192°. Volatile in steam.

*Hydrazide*: needles from  $H_2O$ . M.p. 136°.

*Azide*: yellow plates. M.p. 37°.

*Anilide*: plates. M.p. 140°.

Steinkopf, Ohse, *Ann.*, 1924, 437, 18.

Voerman, *Rec. trav. chim.*, 1907, 26, 293.

Nahnsen, *Ber.*, 1884, 17, 2195.

**$\beta$ -Thiophenic Acid** (*Thiophene-3-carboxylic acid*).

Needles from  $H_2O$ . M.p.  $138.4^\circ$ . Sol. 230 parts  $H_2O$  at  $25^\circ$ . Sublimes. Volatile in steam.  $k = 7.8 \times 10^{-5}$  at  $25^\circ$ . Gives indophenin reaction.

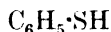
*Amide*: needles from  $Et_2O$ . M.p.  $177.5-178^\circ$ . Spar. sol.  $Et_2O$ .

Voerman, *Rec. trav. chim.*, 1907, **26**, 297.

### **Thiophenin.**

See 2-Aminothiophene.

**Thiophenol** (*Phenyl mercaptan*, *mercapto-benzene*)



$C_6H_6S$

MW, 110

Liq. with penetrating odour. B.p.  $169.5^\circ$ ,  $103.6^\circ/100$  mm.,  $86.2^\circ/50$  mm.,  $77^\circ/30$  mm.  $D_4^{25}$  1.0728,  $D_4^{50}$  1.0491,  $D_4^{75}$  1.0254.  $n_D^{25}$  1.5861. Forms metallic salts. Oxidises in air, especially in alcoholic ammonia solution, to diphenyl disulphide.

*Me ether*: see Thioanisole.

*Et ether*: see Thiophenetole.

*Phenyl ether*: see Diphenyl sulphide.

*Acetyl*: see S-Phenyl ester under Thioacetic Acid.

*Benzoyl*: see S-Phenyl ester under Thio-benzoic Acid.

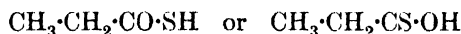
Adams, Marvel, *Organic Syntheses*, Collective Vol. I, 490.

Winter, *Am. Chem. J.*, 1904, **31**, 572.

### **Thiophosgene.**

See Thiocarbonyl chloride.

### **Thiopropionic Acid**



$C_3H_6OS$

MW, 90

Liq. with pungent sulphur-like odour.

*S-Me ester*:  $C_4H_8OS$ . MW, 104. B.p.  $119-20^\circ$ .

*O-Et ester*:  $C_5H_{10}OS$ . MW, 118. B.p.  $130-2^\circ$ .  $D_4^{20}$  0.9451.  $n_D^{20}$  1.46281. Insol.  $H_2O$ .

*Amide*:  $C_3H_7NS$ . MW, 89. Leaflets from  $C_6H_6$ . M.p.  $41-3^\circ$ . Very sol.  $C_6H_6$ . Spar. sol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ .

*Anilide*: needles from  $AcOH.Aq$ . M.p.  $67-67.5^\circ$ .

Delépine, *Bull. soc. chim.*, 1911, **9**, 907.

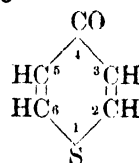
Weigert, *Ber.*, 1903, **36**, 1009.

Hubacher, *Ann.*, 1890, **259**, 229.

### **Thiopropyl Alcohol.**

See Propyl Mercaptan.

### **Thio- $\gamma$ -pyrone**



$C_5H_4OS$

MW, 112

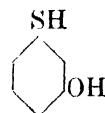
Prisms from  $CCl_4$ . M.p.  $110^\circ$ . Mod. sol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ . Spar. sol. ligroin. Sol. conc.  $H_2SO_4$ .

*HCl salt*: m.p.  $135^\circ$  (not sharp).

*Add. comp. with  $HgCl_2$* : needles from  $H_2O$ . M.p.  $189^\circ$ .

Arndt, Bekir, *Ber.*, 1930, **63**, 2395.

**Thioresorcinol** (*m-Hydroxythiophenol*, *m-mercaptophenol*)



$C_6H_6OS$

MW, 126

Cryst. with penetrating odour. M.p.  $16-17^\circ$ . B.p.  $168^\circ/35$  mm. Sol. common org. solvents except pet. ether. Spar. sol.  $H_2O$ . Volatile in steam.

*O-Me ether*: *m*-mercaptoanisole, *m*-methoxythiophenol.  $C_7H_8OS$ . MW, 140. Liq. B.p.  $224-5^\circ$ ,  $112-14^\circ/20$  mm.,  $96-100^\circ/9-10$  mm.

*S-Me ether*: *m*-hydroxythioanisole, methyl *m*-hydroxyphenyl sulphide. Cryst. M.p.  $15^\circ$ . B.p.  $224^\circ$  slight decomp.,  $148-51^\circ/14$  mm. Sol. common org. solvents except pet. ether. Volatile in steam.

*S-Et ether*: *m*-hydroxythiophenetole, ethyl *m*-hydroxyphenyl sulphide.  $C_8H_{10}OS$ . MW, 154. B.p.  $238-9^\circ$ ,  $104-5^\circ/9-10$  mm.

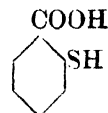
Watson, Dutt, *J. Chem. Soc.*, 1922, **121**, 2415.

Zincke, Ebel, *Ber.*, 1914, **47**, 927.

Szathmáry, *Ber.*, 1910, **43**, 2487.

Mauthner, *Ber.*, 1906, **39**, 3596.

**Thiosalicylic Acid** (*o-Mercaptobenzoic acid*)



$C_7H_6O_2S$

MW, 154

Leaflets or needles from  $EtOH$  or  $AcOH$ . M.p.  $164-5^\circ$ . Sol.  $EtOH$ ,  $AcOH$ . Spar. sol. hot  $H_2O$ . Oxidises in air  $\rightarrow$  diphenyl disulphide 2:2'-dicarboxylic acid.  $FeCl_3$  in alc. sol.  $\rightarrow$  blue col. Alk.  $KMnO_4 \rightarrow$  2-sulphobenzoic acid.



*Me ether* : see S-Methylthiosalicylic Acid.

*Et ether* : S-Ethylthiosalicylic acid.  $C_9H_{10}O_2S$ . MW, 182. Yellowish cryst. M.p. 134–5°. Sol. EtOH, AcOH. Very spar. sol.  $H_2O$ . *Et ester* :  $C_{11}H_{14}O_2S$ . MW, 210. Cryst. M.p. 27–8°. B.p. 152–3°/10 mm.

*Phenyl ether* : see Diphenyl sulphide 2-carboxylic Acid.

*Nitrophenyl ether* : see Nitrodiphenyl sulphide 2-carboxylic Acid.

*p-Tolyl ether* : cryst. from MeOH. M.p. 215–16°.

*2-Naphthyl ether* : leaflets from EtOH. M.p. 200–1°.

*Benzyl ether* : needles. M.p. 189°.

*Acetyl* : thioaspirin. Needles from  $C_6H_6$ . M.p. 125°.

*Me ester* :  $C_8H_8O_2S$ . MW, 168. Oil. B.p. 262–3°/728 mm. Volatile in steam.

*Phenyl ester* : thiosalol.  $C_{13}H_{10}O_2S$ . MW, 230. Cryst. from MeOH. M.p. 91°. Sol. EtOH,  $Et_2O$ . Spar. sol. ligroin.

Allen, Mackay, *Organic Syntheses*, 1932, XII, 76.

Mayer, *Ber.*, 1909, 42, 1134.

### Thiosalol.

See under Thiosalicylic Acid.

### Thiosemicarbazide



$CH_5N_3S$  MW, 91

Needles from  $H_2O$ . M.p. 181–3°.

*Hydrochloride* : m.p. 186–90°.

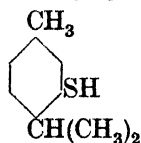
Derivatives are given elsewhere under their own names.

Freund, Schander, *Ber.*, 1896, 29, 2501.

### Thiosinamine.

See Allylthiourea.

**Thiothymol** (3-Mercapto-p-cymene, 3-methyl-6-isopropylthiophenol, thymyl mercaptan)



$C_{10}H_{14}S$  MW, 166

B.p. 230–1°.

$Hg(C_{10}H_{13}S)_2$  : greenish cryst. from EtOH. M.p. 78°.

Fittica, *Ann.*, 1874, 172, 305, 325.

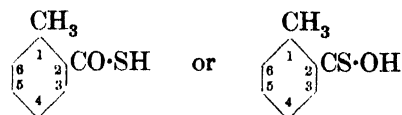
### Thiotolene.

See Methylthiophene.

### Thio-*o*-toluic Acid.

See Phenylthioacetic Acid.

### Thio-*o*-toluic Acid (o-Methylthiobenzoic acid)



$C_8H_8OS$  MW, 152

Yellow oil. B.p. 133°/35 mm.  $D_{25}^{25}$  1.1451. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

*S-Et ester* :  $C_{10}H_{12}OS$ . MW, 180. Yellow oil. B.p. 133°/15 mm.  $D_{25}^{25}$  1.0513. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

*Amide* : o-methylthiobenzamide.  $C_8H_9NS$ . MW, 151. M.p. 88°.

*Phenylhydrazide* : m.p. 116–18°.

Sachs, Reid, *J. Am. Chem. Soc.*, 1916, 38, 2748.

### Thio-*p*-toluic Acid (p-Methylthiobenzoic acid).

Greenish prisms from pet. ether. M.p. 43.5–44°. B.p. 131°/15 mm. Very volatile in steam.

*S-Et ester* : b.p. 150°/18 mm.  $D_{25}^{25}$  1.0708. Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

*O-Et ester* : b.p. 140–5°/70 mm.  $D_4^{20}$  0.9992.

*S-p-Nitrobenzyl ester* : yellow cryst. from EtOH. M.p. 97°.

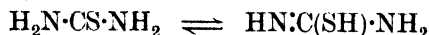
*Amide* : p-methylthiobenzamide. Cryst. M.p. 168°. *N-Benzoyl* : red prisms from EtOH. M.p. 135–6°.

*Anilide* : yellow needles from EtOH. M.p. 140–1°.

*p-Toluidide* : yellow cryst. from  $Me_2CO$ . M.p. 165–6°.

See previous reference.

### Thiourea (Thiocarbamide, thiocarbonic acid diamide)



Isothiourea

$CH_4N_2S$  MW, 76

Rhombohedral or needles from EtOH. M.p. 180°. Sol.  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ . Reacts neutral. Forms add. comps. with metallic salts and oxides. Prolonged heating at 170°  $\rightarrow$   $NH_4CNS$ .  $Ag_2O \rightarrow$  dicyandiamide.

*Hydrochloride* : m.p. 136–7°.

$B_4NH_4Cl$  : cryst. from EtOH. M.p. 154°.

$B_4NH_4Br$  : needles from EtOH. M.p. 180°.

$B_4NH_4I$  : cryst. from EtOH. M.p. 186°.

*S-Me* : see S-Methylisothiurea.

*N* : S-Diacetyl : yellow prisms from AcOH.Aq. M.p. 153°.

*Tri-Me* : prisms from  $C_6H_6$ -ligroin. M.p. 87–8°. Sol.  $H_2O$ , EtOH,  $CHCl_3$ .

N-Tetra-Me : m.p. 78°. B.p. 245°.

N-Tetra-Et : b.p. 264-6°, 130°/12 mm.  
D<sub>4</sub><sup>18</sup> 0.9662. n<sub>D</sub><sup>20</sup> 1.5225.

Triphenyl : needles from EtOH. M.p. 152°.  
Spar. sol. cold EtOH.

N-Tetraphenyl : needles from EtOH. M.p.  
194.5-195.5°. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. Et<sub>2</sub>O. Spar.  
sol. EtOH. Insol. H<sub>2</sub>O.

Other thiourea derivatives are given separately elsewhere.

Kirchoff, Akonjans, *Chem. Zentr.*, 1935,  
I, 2165.

Giua, *Chem. Abstracts*, 1925, **19**, 1561.

I.G., F.P., 655,457, (*Chem. Zentr.*, 1929,  
II, 487).

Ciba, U.S.P., 2,006,762, (*Chem. Abstracts*,  
1935, **29**, 5463).

Werner, *J. Chem. Soc.*, 1912, **101**, 2185.

Schenck, *Z. physiol. Chem.*, 1912, **77**, 370.

Delépine, *Bull. soc. chim.*, 1902, **27**, 814.

I.G., D.R.P., 526,799, (*Chem. Abstracts*,  
1931, **25**, 4892).

Gebhardt, *Ber.*, 1884, **17**, 2092.

### Thiourethane

H<sub>2</sub>N·CS·OC<sub>2</sub>H<sub>5</sub> or H<sub>2</sub>N·CO·SC<sub>2</sub>H<sub>5</sub>

C<sub>3</sub>H<sub>7</sub>ONS MW, 105

O-Ester : Xanthogenamide.

Pyramids. M.p. 40-1° (16°). Decomp. on  
dist. D<sub>4</sub><sup>20</sup> 1.069. n<sub>D</sub><sup>20</sup> 1.520.

N-Di-Me : C<sub>5</sub>H<sub>11</sub>ONS. MW, 133. M.p.  
15°. D<sub>4</sub><sup>20</sup> 1.028. n<sub>D</sub><sup>20</sup> 1.5075.

N-Di-Et : C<sub>7</sub>H<sub>15</sub>ONS. MW, 161. B.p.  
114°/20 mm.

N-Acetyl : prisms from H<sub>2</sub>O. M.p. 101°.

N-Isovaleryl : prisms from Me<sub>2</sub>CO. M.p. 56°.

N-Carbomethoxyl : needles. M.p. 83°.

S-Ester :

Plates. M.p. 102° (109°). Sol. EtOH, Et<sub>2</sub>O,  
hot H<sub>2</sub>O. Insol. cold H<sub>2</sub>O.

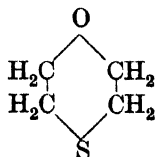
N-Acetyl : prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 98°.

Battegay, Hégazi, *Helv. Chim. Acta*,  
1933, **16**, 1005.

Homberg, *Chem. Abstracts*, 1930, **24**, 2111.

Wheeler, Barnes, *Am. Chem. J.*, 1899,  
**22**, 148.

### Thioxan (1 : 4-Oxthian)



C<sub>4</sub>H<sub>8</sub>OS

MW, 104

M.p. -17°. B.p. 147°, 109.8°/233 mm.,  
86.5°/97 mm., 69.9°/47 mm. D<sub>4</sub><sup>20</sup> 1.1178.  
n<sub>D</sub><sup>20</sup> 1.5081. Spar. sol. H<sub>2</sub>O.

Ethiodide : yellow cryst. from EtOH. M.p.  
85°.

Dibromide : m.p. 75-80° decomp.

Di-iodide : cryst. from Et<sub>2</sub>O. M.p. 66-7°.

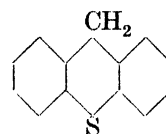
Mercurichloride : needles from EtOH. M.p.  
171°.

Johnson, *J. Chem. Soc.*, 1933, 1530.

Fromm, Ungar, *Ber.*, 1923, **56**, 2288.

Clarke, *J. Chem. Soc.*, 1912, **101**, 1806.

**Thioxanthene** (Diphenylmethane sulphide,  
dibenzthiopyran)



C<sub>13</sub>H<sub>10</sub>S MW, 198

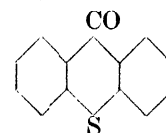
Cryst. from EtOH-CHCl<sub>3</sub>. M.p. 128°. B.p.  
340°/730 mm. Sol. CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O,  
cold EtOH. Sublimes. CrO<sub>3</sub> → benzo-  
phenone sulphone.

S-Oxide : diphenylmethane sulfoxide. Plates  
from pet. ether. M.p. 109-10°.

S-Dioxide : see Diphenylmethane sulphone.

Graebe, Schultefs, *Ann.*, 1891, **263**, 12.

### Thioxanthone (9-Ketothioxanthene)



C<sub>13</sub>H<sub>8</sub>OS MW, 212

Yellow needles from CHCl<sub>3</sub>. M.p. 209° (207°).  
Sublimes. Sol. AcOH, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Insol.  
H<sub>2</sub>O. Red. → thioxanthene. CrO<sub>3</sub> →  
benzophenone sulphone.

S-Dioxide : see Benzophenone sulphone.

Hydrazone : yellow leaflets from EtOH-C<sub>6</sub>H<sub>6</sub>.  
M.p. 115°.

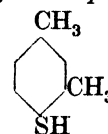
Gomberg, Britton, *J. Am. Chem. Soc.*,  
1921, **43**, 1945.

Graebe, Schultefs, *Ann.*, 1891, **263**, 8.

### Thioxene.

See Dimethylthiophene.

**m-4-Thioxyleneol** (4-Mercapto-m-xylene,  
2 : 4-dimethylphenyl mercaptan)



C<sub>8</sub>H<sub>10</sub>S

MW, 138.

B.p. 207–8° (214°).

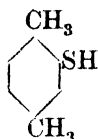
S-*Phenyl*: 2:4-dimethyldiphenyl sulphide.

B.p. 172.5°/11 mm.  $D_4^{15}$  1.0817.

S-p-*Tolyl*: 2:4:4'-trimethyldiphenyl sulphide. B.p. 188°/11 mm.  $D_4^{15}$  1.0614.

Gattermann, *Ber.*, 1899, **32**, 1147.

**p-2-Thioxylenol** (2-Mercapto-p-xylene, 2:5-dimethylphenyl mercaptan)



$C_8H_{10}S$  MW, 138

B.p. 211–12° (205°).

S-*Phenyl*: 2:5-dimethyldiphenyl sulphide.

B.p. 171°/11 mm.  $D_4^{15}$  1.0795.

S-p-*Tolyl*: 2:5:4'-trimethyldiphenyl sulphide. B.p. 185°/11 mm.  $D_4^{15}$  1.0606.

S-*Benzyl*: m.p. 35°. B.p. 200°/15 mm.

See previous reference.

#### Thiuram disulphide

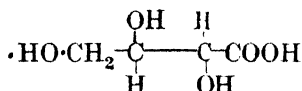


$C_2H_4N_2S_4$  MW, 184

Plates from  $Me_2CO \cdot CHCl_3$ . M.p. 153° decomp. Sol.  $Me_2CO$ , hot EtOH. Insol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ . Heat  $\rightarrow CS_2 + NH_4CNS$ .

Freund, Bachrach, *Ann.*, 1895, **285**, 201.

#### Threonic Acid (Trihydroxybutyric acid)



$C_4H_8O_5$  MW, 136

d-.

Syrup.  $[\alpha]_D$  about  $-30^\circ$  in  $H_2O$ .

*Phenylhydrazide*: laminæ from EtOH. M.p. 157–8°.  $[\alpha]_D^{20} - 29^\circ$  to  $-31^\circ$ .

*Brucine salt*: plates. M.p. 214°.  $[\alpha]_D^{20} - 32.4^\circ$  in  $H_2O$ .

*Quinine salt*: needles from EtOH. M.p. 168°.  $[\alpha]_D^{20} - 116.99^\circ$  in  $H_2O$ .

*Strychnine salt*: plates from dil. EtOH. M.p. 115–21°.  $[\alpha]_D^{20} - 28.5^\circ$  in  $H_2O$ .

l-.

Syrup.  $[\alpha]_D^{15} + 9.54^\circ$ .

*Phenylhydrazide*: m.p. 157°.  $[\alpha]_D^{20}$  about  $+29^\circ$ .

*Me ester*:  $C_5H_{10}O_5$ . MW, 150. *Tri-Me ether*: b.p. 120°/13 mm.  $n_D^{15}$  1.4275.  $[\alpha]_{5780}^{15} + 49^\circ$  in MeOH,  $+31^\circ$  in  $H_2O$ .  $D^{15}$  1.090.

*Amide*:  $C_4H_7O_4N$ . MW, 135. M.p. 88–90°.  $[\alpha]_D^{20} + 58^\circ$  in  $H_2O$ . *Tri-Me ether*: m.p. 78°.  $[\alpha]_{5780}^{20} + 44^\circ$  in  $H_2O$ ,  $+68^\circ$  in MeOH.

*Quinine salt*: m.p. 163–5°

dl-.

M.p. 98–9°.

*Phenylhydrazide*: m.p. 167.5°.

Jensen, Upson, *J. Am. Chem. Soc.*, 1925, **47**, 3023.

Nef, *Ann.*, 1914, **403**, 265.

Nef, Hedenburg, Glattfeld, *J. Am. Chem. Soc.*, 1917, **39**, 1642.

Braun, *J. Am. Chem. Soc.*, 1932, **54**, 1137.

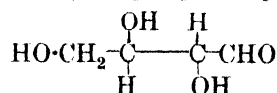
Haworth, Hirst, Smith, *J. Chem. Soc.*, 1934, 1558.

Glattfeld, Hoen, *J. Am. Chem. Soc.*, 1935, **57**, 1407.

Reichstein, Grüssner, Bosshard, *Helv. Chim. Acta*, 1935, **18**, 605.

Herbert et al., *J. Chem. Soc.*, 1933, 1284.

#### Threose (Trihydroxybutyraldehyde)



$C_4H_8O_4$  MW, 120

d-.

M.p. 126–32°. Very hygroscopic.  $[\alpha]_D^{22} + 29.09^\circ \rightarrow 19.59^\circ$  in  $H_2O$ . NaHg  $\rightarrow$  d-erythritol.

*Phenylosazone*: m.p. 164–5°.

*Phenylbenzylhydrazone*: needles from  $C_6H_6$ . M.p. 194.5°.

*Acetone deriv.*: m.p. 84°.  $[\alpha]_D^{22} - 15.27^\circ$  in  $Me_2CO$ .

*Diacetamide deriv.*: prisms. M.p. 166°.  $[\alpha]_D - 10.9^\circ$  in  $H_2O$ .

*Diacetyl*: m.p. 140–2°.  $[\alpha]_D^{23} + 83.52^\circ \rightarrow 34.31^\circ$  in  $CHCl_3$ . Reduces Fehling's.

*Triacetyl*: m.p. 113–14°.  $[\alpha]_D^{20} + 35.5^\circ$  in  $CHCl_3$ .

l-.

Obtained only in solution.  $[\alpha]_D^{15} - 24.6^\circ$  in  $H_2O$ . Reduces Fehling's in the cold.

*Phenylosazone*: cryst. from  $C_6H_6$ . M.p. 165–6°.

*Diacetamide deriv.*: cryst. from 95% EtOH. M.p. 165–6°.

Maquenne, *Ann. chim. phys.*, 1901, **24**, 404.

Ruff, *Ber.*, 1901, **34**, 1370.

Deulofeu, *J. Chem. Soc.*, 1929, 2458; *J. Am. Chem. Soc.*, 1936, **58**, 855.

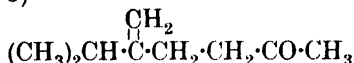
Steiger, Reichstein, *Helv. Chim. Acta*, 1936, **19**, 1016.

Mendive, *Chem. Abstracts*, 1932, **26**, 2433.

Freudenberg, *Ber.*, 1932, **65**, 168.

Hockett, *J. Am. Chem. Soc.*, 1934, **56**, 994.

**Thujaketone** (*Tanacet-ketone*, 2-isopropyl-1-hexenone-5)



$C_9H_{16}O$

MW, 140

B.p. 184–6°.  $D_4^{20}$  0.854.  $n_D^{20}$  1.441.

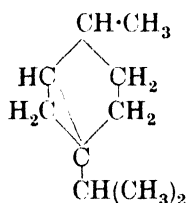
*Oxime*: b.p. 118–20°/15 mm.

*Semicarbazone*: m.p. 143°.

Tiemann, Semmler, *Ber.*, 1897, **30**, 439.

Wallach, *Ann.*, 1893, **272**, 116.

**Thujane** (*Sabinane*)



$C_{10}H_{18}$

MW, 138

*d.*

Mobile oil with faint odour. B.p. 157°/758 mm.  $D_4^{20}$  0.8139.  $n_D^{20}$  1.43759.  $[\alpha]_D + 62.03^\circ$ .

Tschugaev, Fomin, *Compt. rend.*, 1910, **151**, 1058.

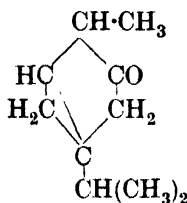
Henderson, Robertson, *J. Chem. Soc.*, 1923, **123**, 1715.

Zelinsky, Turowa-Pollak, *Ber.*, 1929, **62**, 2868.

**Thujol.**

See Thujyl Alcohol.

**Thujone**



$C_{10}H_{16}O$

MW, 152

Two stereoisomers are known.

$\alpha$ -.

Constituent of many essential oils. Oil with fresh odour. B.p. 199–201°, 103–4°/40 mm.  $D_4^{20}$  0.9152.  $n_D^{20}$  1.4530.  $[\alpha]_D^{20} - 11.58^\circ$ . Heat  $\rightarrow$  carvacrol + carvotanacetone. Boiling alc.  $H_2SO_4 \rightarrow \beta$ -thujone  $\rightarrow$  isothujone.

*Oxime*: liq.  $[\alpha]_D^{19} - 29.25^\circ$  in  $Et_2O$ .

*Semicarbazone*: (i) Prisms from MeOH. M.p. 186–8°.  $[\alpha]_D + 64.4^\circ$  in EtOH. (ii) Amorphous. M.p. 100–10°.  $[\alpha]_D^{20} + 53.71^\circ$ .

2:4-Dinitrophenylhydrazone: orange-yellow plates from EtOH. M.p. 116–17°.

*m*-Nitrobenzoylhydrazone: m.p. 156–156.3°.

$\beta$ -. Tanacetone.

Constituent of many essential oils. Oil with odour resembling menthol. B.p. 201–2°.  $D_4^{15.1}$  0.9193.  $n_D^{13.6}$  1.4540.  $[\alpha]_D^{20} + 77.33^\circ$ . Heat of comb.  $C_v$  1429.9 Cal. Alc. KOH  $\rightarrow \alpha$ -thujone. Heat  $\rightarrow$  carvotanacetone. Boiling dil.  $H_2SO_4 \rightarrow$  isothujone. Na + EtOH  $\rightarrow$  thujyl alcohol. Forms cryst. bisulphite comp.

*Oxime*: tanacetone oxime. Prisms. M.p. 54–5°. B.p. 135–6°/20 mm.  $[\alpha]_D^{11} + 105.1^\circ$  in MeOH. *Benzoyl*: cryst. from MeOH. M.p. 52–3°.

*Hydrazone*: b.p. 149°/35 mm.  $D_4^{22}$  0.9504.  $n_D^{22}$  1.4952.  $[\alpha]_D + 123.75^\circ$ .

*Semicarbazone*: (i) Hexagonal cryst. M.p. 174–5°.  $[\alpha]_D^{15} + 218.04^\circ$  in MeOH. (ii) Rhombic cryst. M.p. 170–2°.

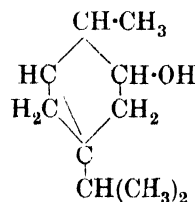
Challenger, *Industrial Chemist*, 1928, **4**, 315.

Paolini, *Chem. Abstracts*, 1926, **20**, 1072.

Östling, Roth, *Ber.*, 1913, **46**, 313.

Rose, Livingstone, *J. Am. Chem. Soc.*, 1912, **34**, 201.

**Thujyl Alcohol** ( $\beta$ -Thujyl alcohol, tanacetyl alcohol, thujol)



$C_{10}H_{18}O$

MW, 154

Eight active and four racemic isomers are possible. Occurs free and combined as ester in several essential oils. Characteristic constants for the commercial product (mixture of isomers) are: b.p. 208–10° (220°), 92.5°/13 mm.  $D_4^{25}$  0.9266.  $n_D^{25}$  1.4621. Heat of comb.  $C_v$  1477.1 Cal.

*Me ether*:  $C_{11}H_{20}O$ . MW, 168.  $D_4^{20}$  0.8771.  $n_D$  1.44541.

Two dextro- and one laevorotatory modifications are described in the literature.

*d.*  $\beta$ -Thujyl alcohol.

B.p. 206°.  $D_4^{20}$  0.9187.  $n_D^{16}$  1.4625.  $[\alpha]_D^{20} + 116.93^\circ$ .  $CrO_3 \rightarrow \beta$ -thujone.

*Hydrogen phthalate*: needles from pet. ether. M.p. 120°.  $[\alpha]_D + 91.3^\circ$  in EtOH. *Ag salt*: m.p. 85–6°. *Stychnine salt*: needles from EtOH.Aq. M.p. 177–8°.  $[\alpha]_D + 36.8^\circ$ .

*d*-.  $\delta$ -Thujyl alcohol.

B.p. 206°.  $n_D^{20}$  1.4759.  $[\alpha]_D^{20} + 50.01^\circ$ .

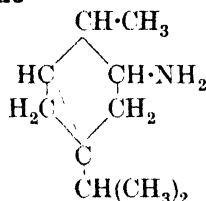
*Hydrogen phthalate*: cryst. from pet. ether.  
M.p. 95-6°.  $[\alpha]_D^{20} + 2.3^\circ$  in EtOH.

*l*-.  
M.p. 28°.  $[\alpha]_D^{20} - 9.12^\circ$  in toluene.

Tschugajew, Fomin, *Ber.*, 1912, **45**, 1295.  
Paolini, Lomonaco, *Atti accad. Lincei*,  
1914, **23**, (11), 128.

Rose, Livingstone, *J. Am. Chem. Soc.*,  
1912, **34**, 202.

### Thujylamine



$C_{10}H_{19}N$

MW, 153

As with thujyl alcohol, eight active and four racemic modifications are possible. Three thujylamines are described but the constitution of one only is substantiated.

$\alpha$ -.  
B.p. 198-9°.

$\beta$ -. From  $\beta$ -thujone.

B.p. 198-205°, 75-90°/14 mm.  $D_4^{20}$  0.876.  
 $n_D^{20}$  1.46782.  $[\alpha]_D^{20} + 22.34^\circ$ . Dist. hydrochloride  
→ isothujene.

*B.HCl*: m.p. 210-14°.  $[\alpha]_D^{20} + 32.67^\circ$  in  
EtOH.

$\beta$ -. From  $\beta$ -thujone-oxime. Tanacetylamine.

B.p. 195°, 80.5°/14 mm.  $D_4^{20}$  0.8712.  $n_D^{20}$   
1.4608.  $[\alpha]_D^{20} + 101.06^\circ$ . Dist. hydrochloride  
→ isothujene.

*B.HCl*: m.p. 260-1°.

*Nitrate*: cryst. from  $H_2O$ . M.p. 167-8°.  
 $[\alpha]_D^{20} + 82.03^\circ$ .

*Carbonate*: m.p. 106-7°.

*N-Di-Me*:  $C_{12}H_{23}N$ . MW, 181. B.p. 213.5-  
214°.  $D_4^{20}$  0.8606.  $[\alpha]_D^{20} + 141.76^\circ$ .

Kondakow, Skworzow, *J. prakt. Chem.*,  
1904, **69**, 178.

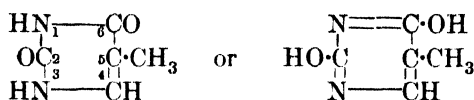
Tschugajew, *Ber.*, 1901, **34**, 2278.

Wallach, *Ann.*, 1893, **272**, 109.

### Thymacetin.

See under 6-Aminothymol.

### Thymine (5-Methyluracil)



$C_5H_6O_2N_2$

MW, 126

Plates from  $H_2O$ . M.p. 326° (318-20°). Sol.  
250 parts  $H_2O$  at 25°. Spar. sol. EtOH,  $Et_2O$ .  
Sublimes. Heat of comb.  $C_7$  566.4 Cal.

1-*N-Me*: 1:5-dimethyluracil.  $C_6H_8O_2N_2$ .  
MW, 140. Prisms from  $H_2O$ . M.p. 202-5°  
decomp. Very sol. hot EtOH,  $Me_2CO$ .

3-*N-Me*: 3:5-dimethyluracil. Needles or  
prisms from  $H_2O$ . M.p. 280-2°.

1:3-*N-Di-Me*: 1:3:5-trimethyluracil.  
 $C_7H_{10}O_2N_2$ . MW, 154. Needles from EtOH.  
M.p. 153°. Very sol.  $H_2O$ ,  $CHCl_3$ . Spar. sol.  
 $Et_2O$ , pet. ether.

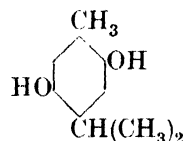
Bergmann, Currie, *J. Am. Chem. Soc.*,  
1933, **55**, 1734.

Johnson, Clapp, *J. Biol. Chem.*, 1908,  
**5**, 56.

### Thyminose.

See 2-Ribodeseose.

**Thymohydroquinone** (2:5-Dihydroxy-p-  
cymene, thymoquinol, hydrothymoquinone)



$C_{10}H_{14}O_2$

MW, 166

Occurs free and combined as ether in several  
essential oils. Prisms. M.p. 143° (139.5°). B.p.  
290°. Very sol. EtOH,  $Et_2O$ . Sol. hot  $H_2O$ .  
Insol.  $C_6H_6$ . Sublimes. Heat of comb.  $C_7$   
1308.6 Cal.,  $C_8$  1308.1 Cal. Ox. → thymo-  
quinone.

*Di-Me ether*:  $C_{12}H_{18}O_2$ . MW, 194. B.p. 248-  
50°, 118°/12 mm.  $D_4^{20}$  0.998.  $n_D^{20}$  1.51339.

*Diacetyl*: m.p. 73-5°.

*Dibenzoyl*: pale yellow needles from EtOH.  
M.p. 141-2°.

*Phenylurethane*: leaflets from EtOH. M.p.  
232-3°.

$\alpha$ -*Naphthylurethane*: cryst. from EtOH. M.p.  
147-8°.

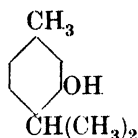
Sherk, *Chem. Zentr.*, 1921, III, 218.

Sabatier, Mailhe, *Compt. rend.*, 1908, **146**,  
458.

### Thymohydroxycuminic Acid.

See 3-Hydroxycuminic Acid.

**Thymol** (3-Hydroxy-p-cymene, 3-hydroxy-4-isopropyltoluene, 3-methyl-6-isopropylphenol, 4-isopropyl-m-cresol)



$C_{10}H_{14}O$

MW, 150

Constituent of numerous essential oils. Plates with odour of thyme from AcOEt, AcOH or Me<sub>2</sub>CO. M.p. 51.5°. B.p. 233.5°. Sol. 1176 parts H<sub>2</sub>O at 19.4°. Very sol. EtOH, Et<sub>2</sub>O, AcOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Heat of comb.  $C_p$  1353.75 Cal. Crit. temp. 425.1°. Red.  $\rightarrow$  menthol + menthone. Ox.  $\rightarrow$  thymohydroquinone or dithymol. KNO<sub>2</sub> + H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  green  $\rightarrow$  blue col. Possesses antiseptic properties.

*Me ether*: C<sub>11</sub>H<sub>16</sub>O. MW, 164. Occurs naturally. Liq. with ethereal odour. B.p. 211–12°/745 mm., 94–6°/15 mm.  $D_4^{20}$  0.9388.

*Et ether*: C<sub>12</sub>H<sub>18</sub>O. MW, 178. B.p. 224–8°.  $D_0^0$  0.9334.

*Propyl ether*: C<sub>13</sub>H<sub>20</sub>O. MW, 192. B.p. 243°.  $D_0^0$  0.9276.

*Butyl ether*: C<sub>14</sub>H<sub>22</sub>O. MW, 206. B.p. 258.3°.  $D_0^0$  0.9230.

*d-Amyl ether*: C<sub>15</sub>H<sub>24</sub>O. MW, 220. B.p. 250–60°.  $D_0^{18}$  0.934.  $n_D^{17}$  1.5056.  $[\alpha]_D^{18} + 4.17^\circ$ .

*Isoamyl ether*: b.p. 242–3°/746.5 mm.  $n_D^{15}$  1.4923.

*Phenyl ether*: C<sub>16</sub>H<sub>18</sub>O. MW, 226. B.p. 297°/766 mm., 176°/25 mm.  $D_0^{15}$  1.0113.

*Benzyl ether*: C<sub>17</sub>H<sub>20</sub>O. MW, 240. B.p. 221–3°/35 mm.  $D_0^{18}$  1.0063.  $n_D^{20}$  1.5511.

*p-Nitrobenzyl ether*: C<sub>17</sub>H<sub>15</sub>O<sub>3</sub>N. MW, 285. Cryst. from EtOH.Aq. M.p. 85.5°.

*Formyl*: b.p. 81°/2 mm.  $D_0^0$  1.015.  $n_D^{20}$  1.49606.

*Acetyl*: b.p. 242–3°.  $D_0^0$  1.009.

*Oxalate*: needles from EtOH. M.p. 61°.

*Benzoyl*: plates. M.p. 33°.

*3:5-Dinitrobenzoyl*: needles. M.p. 103.2°.

*Phenylurethane*: cryst. from EtOH.Aq. M.p. 106.5–107°.

$\alpha$ -Naphthylurethane: m.p. 160°.

Rheinische Kampher-Fabrik., E.P., 308,681 (*Chem. Zentr.*, 1931, II, 1492). Austerweil, Lemay, *Bull. soc. chim.*, 1927, 41, 454.

Frisch, D.R.P., 615,470, (*Chem. Zentr.*, 1936, 107, I, 883; *Chem. Abstracts*, 1935, 29, 6252).

Hund, U.S.P., 1,967,440.

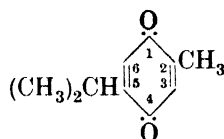
**Thymomenthone.**

See under Menthone.

**Thymoquinol.**

See Thymohydroquinone.

**Thymoquinone** (1-Methyl-4-isopropylcyclohexadiene-3:6-dione, 2-methyl-5-isopropyl-1:4-benzoquinone)



$C_{10}H_{12}O_2$

MW, 164

Occurs in oil from wood of *Callitris quadrivalvis*, Vent., and *Monarda fistulosa*. Bright yellow tablets with penetrating odour. M.p. 45.5°. B.p. 232°. Very sol. EtOH, Et<sub>2</sub>O. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Volatile in steam. Heat of comb.  $C_p$  1274.6 Cal.,  $C_r$  1273.4 Cal. In light  $\rightarrow$  dithymoquinone. EtOH + light  $\rightarrow$  thymohydroquinone. Red.  $\rightarrow$  thymohydroquinone.

*Thymohydroquinone add. comp.*: dark violet needles. M.p. 64°.

*1-Oxime*: see 6-Nitrosothymol.

*4-Oxime*: see 5-Nitrosocarvacrol.

*1:4-Dioxime*: C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub>. MW, 194. Prisms from EtOH. Darkens at 200°. Decomp. at 235°. *Diacetyl*: plates or needles from ligroin. M.p. 110°. *Dibenzoyl*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 199–200°.

*1-Semicarbazone*: yellow needles from EtOH. M.p. 201–2° decomp.

*1:4-Disemicarbazone*: cryst. from AcOH. M.p. 237°.

*1-Phenylsemicarbazone*: dark yellow needles from AcOH. M.p. 204–5°.

*1:4-Diphenylsemicarbazone*: dark yellow cryst. powder. Decomp. at 242°.

*1-Phenylhydrazone*: reddish-yellow needles from EtOH. M.p. 93°.

*1-o-Nitrophenylhydrazone*: red needles from MeOH. M.p. 145°.

*1-[2:4-Dinitrophenyl]-hydrazone*: dark red needles from EtOH. M.p. 179–80°.

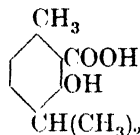
Tseng, Hu, Chu, *J. Chinese Chem. Soc.*, 1934, 2, 151.

Kremers, Hixon, Wakeman, *Organic Syntheses*, 1926, VI, 92.

Liebermann, Iljinski, *Ber.*, 1885, 18, 3194, 3220.

**o-Thymotinic Acid** (3-Hydroxy-4-isopropyl-o-toluic acid, 3-hydroxy-1-methyl-4-isopropyl-

benzene-2-carboxylic acid, 6-methyl-3-isopropyl-salicylic acid)



$C_{11}H_{14}O_3$

MW, 194

Needles from  $H_2O$ ,  $C_6H_6$  or ligroin. M.p.  $127^\circ$ . Sol. 10,000 parts  $H_2O$  at  $20^\circ$ . Sol. EtOH,  $Et_2O$ , AcOH,  $CHCl_3$ ,  $C_6H_6$ . Volatile in steam.  $FeCl_3 \rightarrow$  dark blue col. Strong bactericide.

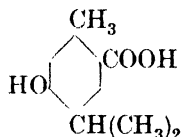
Me ester:  $C_{12}H_{16}O_3$ . MW, 208. Yellow oil. B.p.  $142^\circ/18.5$  mm.

Et ester:  $C_{13}H_{18}O_3$ . MW, 222. Yellow oil. B.p.  $153^\circ/18.5$  mm.

Acetonyl ester:  $C_{14}H_{18}O_4$ . MW, 250. Needles from EtOH. M.p.  $75^\circ$ . Very sol. hot EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Has local anaesthetic action.

Spallino, Provenzal, *Gazz. chim. ital.*, 1909, **39**, 326.

**p-Thymotinic Acid** (5-Hydroxy-4-isopropyl-o-toluic acid, 5-hydroxy-1-methyl-4-isopropyl-benzene-2-carboxylic acid)



$C_{11}H_{14}O_3$

MW, 194

Plates from EtOH.Aq. M.p.  $157^\circ$ . Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. hot  $H_2O$ . No col. with  $FeCl_3$ .

Me ether: 2-methyl-5-isopropylanisic acid.  $C_{12}H_{16}O_3$ . MW, 208. Needles from EtOH. M.p.  $138-9^\circ$ . Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. cold  $H_2O$ . Cu salt: blue cryst. from EtOH-ligroin. Decomp. at  $190^\circ$ . Et ester:  $C_{14}H_{20}O_3$ . MW, 236. Liq. with unpleasant odour. B.p.  $163-4^\circ/13$  mm.  $D_4^{20}$  1.032. Amide:  $C_{12}H_{17}O_2N$ . MW, 207. Needles from EtOH. M.p.  $158-9^\circ$ . Sol. EtOH, AcOH. Spar. sol.  $Et_2O$ , ligroin. Nitrile:  $C_{12}H_{15}ON$ . MW, 189. Cryst. from ligroin. M.p.  $69-70^\circ$ . B.p.  $158-60^\circ/16$  mm. Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Sol. ligroin.

Et ether:  $C_{13}H_{18}O_3$ . MW, 222. Prisms from EtOH. M.p.  $159^\circ$ . Spar. sol.  $H_2O$ . Amide:  $C_{13}H_{19}O_2N$ . MW, 221. Needles from EtOH. M.p.  $127^\circ$ .

Me ester:  $C_{12}H_{16}O_3$ . MW, 208. Cryst. from  $Et_2O$  or  $C_6H_6$ . M.p.  $97-8^\circ$ .

Nitrile:  $C_{11}H_{13}ON$ . MW, 175. Cryst. from  $C_6H_6$ . M.p.  $115-16^\circ$ .

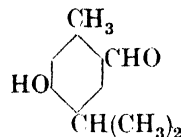
Houben, Fischer, *Ber.*, 1931, **64**, 245.

Grignard, Bellet, Courtot, *Ann. chim.*, 1915, **4**, 50.

Gattermann, *Ann.*, 1888, **244**, 69.

Kobek, *Ber.*, 1883, **16**, 2102.

**p-Thymotinic Aldehyde** (5-Hydroxy-4-isopropyl-o-toluic aldehyde, 6-aldehydothymol, 4-hydroxy-2-methyl-5-isopropylbenzaldehyde)



$C_{11}H_{14}O_2$

MW, 178

Needles from hot  $H_2O$ . M.p.  $133^\circ$ . Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. hot  $H_2O$ . Forms cryst. bisulphite comp. No col. with  $FeCl_3$ .

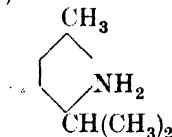
Me ether: 2-methyl-5-isopropylanisaldehyde.  $C_{12}H_{16}O_2$ . MW, 192. B.p.  $278^\circ$ . Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Anil: plates from ligroin. M.p.  $80^\circ$ .

Anil: pale yellow needles. M.p.  $142^\circ$ .

Gattermann, Berchemann, *Ber.*, 1898, **31**, 1767.

Kobek, *Ber.*, 1883, **16**, 2097.

**Thymylamine** (5-Methyl-2-isopropylaniline, 3-amino-p-cymene)



$C_{10}H_{15}N$

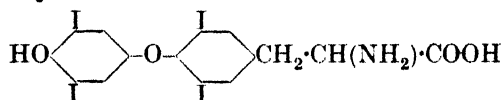
MW, 149

Oil with unpleasant odour. B.p.  $230^\circ$ . Volatile in steam. Misc. with EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

Acetyl: needles from EtOH. M.p.  $112^\circ$ .

Lloyd, *Ber.*, 1887, **20**, 1259.

**Thyroxine**



$C_{15}H_{11}O_4NI_4$

MW, 777

dl-.

Isolated from thyroid gland. Needles by addition of AcOH to alk. EtOH.Aq. sol. Darkens at  $220^\circ$ . M.p.  $231-3^\circ$  ( $250^\circ$ ) decomp. Very spar. sol.  $H_2O$ . Insol. org. solvents. Sol.  $NH_3$ .

and alc. alkalis. Increases rate of basal metabolism.

*Me ester*:  $C_{16}H_{13}O_4NI_4$ . MW, 791. Prisms from EtOH.Aq. M.p. 156°. Spar. sol.  $H_2O$ , org. solvents except EtOH. *B.HCl*: needles from EtOH-HCl. M.p. 221-5°. *N-Acetyl*: plates from anisole. M.p. 208-9° decomp. *N-Chloroacetyl*: prisms from  $C_6H_6$ . M.p. 159-60°.

*Et ester*:  $C_{17}H_{15}O_4NI_4$ . MW, 805. *N-Diacetyl*: needles. M.p. 216-17° (230°).

*N-Acetyl*: cryst. from AcOH.Aq. M.p. 210-15° decomp.

*N-Chloroacetyl*: cryst. from AcOH.Aq. M.p. 201-2° decomp.

*l.*

M.p. 235°.  $[\alpha]_{D_{461}}^{21} - 4.45^\circ$  in EtOH-NaOH.

*d.*

$[\alpha]_{D_{461}}^{21} + 2.97^\circ$  in EtOH-NaOH.

Harington, Slater, *Biochem. J.*, 1930, **24**, 456.

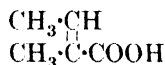
*Thyroxine*, Kendall, (Chem. Catalog. Co., New York, 1929).

Ashley, Harington, *Biochem. J.*, 1929, **23**, 1178; 1928, **22**, 1436.

Harington, *Biochem. J.*, 1928, **22**, 1429; 1926, **20**, 293.

Harington, Barger, *Biochem. J.*, 1927, **21**, 169.

**Tiglic Acid** (cis-1 : 2-Dimethylacrylic acid, 1-methylcrotonic acid, 1-ethylidenepropionic acid)



$C_5H_8O_2$

MW, 100

Occurs as glyceride in croton oil, as ester in Roman cummin oil. Tablets and columns from  $H_2O$ . M.p. 64°. B.p. 198-5°.  $D_4^{20}$  0.9641.  $n_D^{76}$  1.43297. Sol. hot  $H_2O$ . Heat of comb. 626.6 Cal.  $k = 0.957 \times 10^{-5}$  at 25°.  $HI (+ P) \longrightarrow$  1-methylbutyric acid.

*Me ester*:  $C_6H_{10}O_2$ . MW, 114. B.p. 139-4-139.6°/766 mm.  $D_4^{20}$  0.94980.  $n_D^{20}$  1.43700.

*Et ester*:  $C_7H_{12}O_2$ . MW, 128. B.p. 156°/752 mm., 80.5-81.5°/45 mm., 55.5°/11 mm.  $D_4^{19.5}$  0.9247.  $n_D^{19.5}$  1.43554.

*Isoamyl ester*:  $C_{10}H_{18}O_2$ . MW, 170. Occurs in Roman cummin oil. B.p. 204-5°.

*d-Citronellyl ester*:  $C_{15}H_{26}O_2$ . MW, 238. Liq. with pleasant odour. B.p. 144-5°/7 mm.  $D_{15}^{15}$  0.9090.

*Geranyl ester*:  $C_{15}H_{24}O_2$ . MW, 236. Liq. with pleasant odour. B.p. 149-51°/7 mm.  $D_{15}^{15}$  0.9279.

*p-Bromophenacyl ester*: cryst. from EtOH. M.p. 67-9°.

*Chloride*:  $C_5H_7OCl$ . MW, 118.5. B.p. 45°/12 mm.

*Anilide*: cryst. from pet. ether. M.p. 77°.

*2-Naphthylamide*: cryst. M.p. 96°.

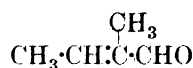
Michael, Ross, *J. Am. Chem. Soc.*, 1933, **55**, 3692.

Auwers, Wissebach, *Ber.*, 1923, **56**, 715.

Auwers, *Ann.*, 1923, **432**, 70.

Blaise, *Bull. soc. chim.*, 1903, **29**, 330.

**Tiglic Aldehyde** (1 : 2-Dimethylacrolein)



$C_5H_8O$

MW, 84

Liq. with penetrating odour. B.p. 116-5-117.5°/738 mm., 63-2-65.0°/119 mm.  $D_4^{20}$  0.8710.  $n_D^{20}$  1.4475. Sol. 40-50 parts  $H_2O$ . Misc. with EtOH,  $Et_2O$ . Oxidises in air. Forms cryst. bisulphite comp.

*Oxime*: cryst. from  $Et_2O$ . M.p. 43°. B.p. 66-7°/10 mm.

*Semicarbazone*: needles from EtOH. M.p. 219° (234°).

*Phenylhydrazone*: needles from petrol. M.p. 92-4°. B.p. 163-8°/17 mm., 155-60°/9 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. petrol, pet. ether.

*p-Nitrophenylhydrazone*: cryst. from EtOH.Aq. M.p. 181°.

*2 : 4-Dinitrophenylhydrazone*: red cryst. from AcOEt-Me<sub>2</sub>CO. M.p. 206-7° (222°).

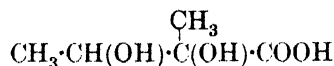
Pummerer, Reindel, *Ber.*, 1933, **66**, 335.

Shepard, Johnson, *J. Am. Chem. Soc.*, 1932, **54**, 4390.

Auwers, Krender, *Ber.*, 1925, **58**, 1978.

Grignard, Ablemann, *Bull. soc. chim.*, 1910, **7**, 643.

**Tigliceric Acid** (1 : 2-Dimethylglyceric acid)



$C_5H_{10}O_4$

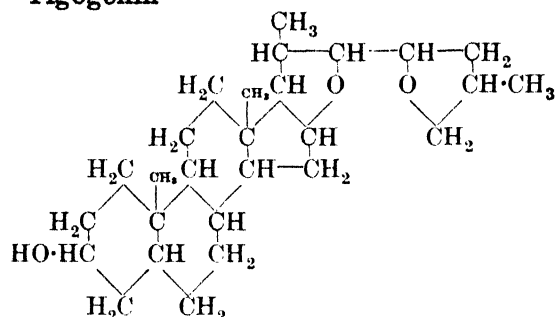
MW, 134

Stereoisomeric with angliceric acid. Prisms from  $Et_2O$ . M.p. 88°. Very sol.  $H_2O$ , EtOH, Me<sub>2</sub>CO. Spar. sol. cold  $Et_2O$ . Insol.  $CHCl_3$ ,  $C_6H_6$ , ligroin. Most salts sol.  $H_2O$ .

Fittig, Penschuck, *Ann.*, 1894, **283**, 109.



## Tigogenin

 $C_{27}H_{44}O_3$ 

MW, 416

Occurs in *Digitalis purpurea*, *D. lanata*, *Chlorogalum pomeridianum*. Cryst. +  $1H_2O$  from EtOH. M.p. 205–6°. Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, pet. ether.  $[\alpha]_D^{25} - 67.2^\circ$  in CHCl<sub>3</sub>.

Acetyl: plates from MeOH. M.p. 202°.  $[\alpha]_D^{25} - 74.4^\circ$  in CHCl<sub>3</sub>.

Benzoyl: cryst. from MeOH-CHCl<sub>3</sub>. M.p. 230–3°.  $[\alpha]_D^{22.5} - 68^\circ$  in CHCl<sub>3</sub>.

o-Bromobenzoyl: cryst. from EtOH-CHCl<sub>3</sub>. M.p. 210–12°.  $[\alpha]_D^{27} - 56^\circ$  in CHCl<sub>3</sub>.

Jacobs, Fleck, *J. Biol. Chem.*, 1930, **88**, 545.

Liang, Noller, *J. Am. Chem. Soc.*, 1935, **57**, 526.

Tschesche, Hagedorn, *Ber.*, 1935, **68**, 1416, 2247.

## Tigonin

 $C_{56}H_{92}O_{27}$ 

MW, 1196

Glucoside present in leaves of *Digitalis lanata*. Amorph. from EtOH. Sinters at 220°. M.p. 260°. Absorbs 2H<sub>2</sub>O in moist air. Forms add. comp. with cholesterol. Hyd. → tigogenin + galactose + glucose + xylose.

Cholesterol add. comp.: needles from MeOH. Decomp. above 200°.

Tschesche, *Ber.*, 1936, **69**, 1665.

## Tin diethyl

 $Sn(C_2H_5)_2$  $C_4H_{10}Sn$ 

MW, 177

Yellow oil. Decomp. on dist. → Sn + tin tetra-ethyl. Sol. EtOH, Et<sub>2</sub>O, ligroin, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. Oxidises in air → diethyl tin oxide. Cl → Et<sub>2</sub>SnCl<sub>2</sub>. Reduces AgNO<sub>3</sub>.

Pfeiffer, *Ber.*, 1911, **44**, 1270.

## Tin tetra-n-amyl

 $Sn(CH_2 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot CH_3)_4$  $C_{20}H_{44}Sn$ 

MW, 403

Colourless stable liq. B.p. 181°/10 mm.  $D_4^{20}$  1.0206.  $n_D^{20}$  1.4720. Heat of comb. 3384 Cal.

Jones, Evans, Gulwell, Griffiths, *J. Chem. Soc.*, 1935, 41.

## Tin tetra-active-amyl

$$Sn(CH_2 \cdot \overset{\text{CH}_3}{\underset{|}{CH}} \cdot CH_2 \cdot CH_3)_4$$
 $C_{20}H_{44}Sn$ 

MW, 403

Colourless stable liq. B.p. 174°/10 mm.  $D_4^{20}$  1.0222.  $n_D^{20}$  1.4730.

See previous reference.

## Tin tetrabenzyl

 $Sn(CH_2 \cdot C_6H_5)_4$  $C_{28}H_{28}Sn$ 

MW, 483

Prisms from pet. ether. M.p. 42–3°. Very sol. most org. solvents. Spar. sol. pet. ether. Oxidises in air → benzaldehyde.

Smith, Kipping, *J. Chem. Soc.*, 1912, **101**, 2559.

## Tin tetra-n-butyl

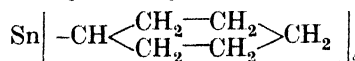
 $Sn(CH_2 \cdot CH_2 \cdot CH_2 \cdot CH_3)_4$  $C_{16}H_{36}Sn$ 

MW, 347

Colourless stable liq. B.p. 145°/10 mm.  $D_4^{20}$  1.0572.  $n_D^{20}$  1.4730. Heat of comb. 2773 Cal.

Jones, Evans, Gulwell, Griffiths, *J. Chem. Soc.*, 1935, 41.

## Tin tetracyclohexyl

 $C_{24}H_{44}Sn$ 

MW, 451

Plates from C<sub>6</sub>H<sub>6</sub>. M.p. 263–4°. Very sol. hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. hot EtOH, warm Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

Krause, Pohland, *Ber.*, 1924, **57**, 535.

## Tin tetraethyl

 $Sn(C_2H_5)_4$  $C_8H_{20}Sn$ 

MW, 235

Colourless stable liq. F.p. – 112°. B.p. 175°, 73°/10 mm.  $D_4^{19.7}$  1.1988.  $n_D^{19.7}$  1.4724. Sol. Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Heat of comb. 1521 Cal.

Emmert, Eller, *Ber.*, 1911, **44**, 2331.

Pfeiffer, Schnurmann, *Ber.*, 1904, **37**, 320.

## Tin tetraisoamyl

 $Sn(CH_2 \cdot CH_2 \cdot CH(CH_3)_2)_4$  $C_{20}H_{44}Sn$ 

MW, 403

Liq. B.p. 188°/24 mm.  $D_4^{19.6}$  1.0353.  $n_D^{19}$  1.4724.

Krause, *Ber.*, 1918, **51**, 1456.

## Tin tetraisobutyl

$\text{Sn}(\text{CH}_2\cdot\text{CH}(\text{CH}_3)_2)_4$   
 $\text{C}_{16}\text{H}_{36}\text{Sn}$  MW, 347  
 Needles. M.p.  $-13^\circ$ . B.p.  $143^\circ/16.5$  mm.  
 $D_4^{25}$  1.0540.  $n_D^{25}$  1.4742.

Grüttner, Krause, *Ber.*, 1917, **50**, 1806.

## Tin tetramethyl

$\text{Sn}(\text{CH}_3)_4$   
 $\text{C}_4\text{H}_{12}\text{Sn}$  MW, 179  
 Liq. with ethereal odour. B.p.  $78^\circ$ .  $D_4^{25}$  1.29136.  $n_D$  1.52009. Insol.  $\text{H}_2\text{O}$ . Reduces alc.  $\text{AgNO}_3$ .

Kraus, Callis, U.S.P., 1,639,947, (*Chem. Abstracts*, 1927, **21**, 3180).

Cahours, *Ann.*, 1860, **114**, 372.

## Tin tetraphenyl

$\text{Sn}(\text{C}_6\text{H}_5)_4$   
 $\text{C}_{24}\text{H}_{20}\text{Sn}$  MW, 427  
 Prisms from  $\text{CHCl}_3$  or dipropylamine, needles from Py. M.p.  $225.7^\circ$ . Very sol. at the boil in  $\text{CHCl}_3$ ,  $\text{AcOH}$ ,  $\text{CS}_2$ , Py,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol. pet. ether.

Chambers, Scherer, *J. Am. Chem. Soc.*, 1926, **48**, 1054.

Emmert, Eller, *Ber.*, 1911, **44**, 2331.

Pope, Peachey, *Chem. News*, 1904, **89**, 20.

## Tin tetrapropyl

$\text{Sn}(\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_3)_4$   
 $\text{C}_{12}\text{H}_{28}\text{Sn}$  MW, 291  
 Colourless stable liq. B.p.  $228^\circ$ ,  $112^\circ/10$  mm.  
 Heat of comb. 2163 Cal.

Pfeiffer, *Z. anorg. Chem.*, 1910, **68**, 121.

Tin tetra-*o*-tolyl

$\text{Sn}\left[\begin{array}{c} \text{CH}_3 \\ \diagup \quad \diagdown \\ \text{C}_6\text{H}_4 \end{array}\right]_4$   
 $\text{C}_{28}\text{H}_{28}\text{Sn}$  MW, 483  
 Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $214-15^\circ$ . Very sol.  $\text{C}_6\text{H}_6$ . Sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{EtOH}$ .

Krause, Becker, *Ber.*, 1920, **53**, 185.

Krause, Schmitz, *Ber.*, 1919, **52**, 2158.

Tin tetra-*p*-tolyl

$\text{Sn}\left[\begin{array}{c} \diagdown \quad \diagup \\ \text{C}_6\text{H}_4 \end{array}\right]_4$   
 $\text{C}_{28}\text{H}_{28}\text{Sn}$  MW, 483  
 Needles from Py. M.p.  $230^\circ$ . Very sol. hot  $\text{AcOEt}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ , hot Py. Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{EtOH}$ , hot  $\text{AcOH}$ .

Pfeiffer, *Z. anorg. Chem.*, 1910, **68**, 122.

Dict. of Org. Comp.—III.

## T.N.T.

See 2 : 4 : 6-Trinitrotoluene.

## Tobias Acid.

See 2-Naphthylamine-1-sulphonic Acid.

$\alpha$ -Tocopherol (*Anti-sterility vitamin*, *vitamin E*)

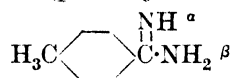
$\text{C}_{29}\text{H}_{50}\text{O}_2$  (?) MW, 430 (?)

$\alpha$ -Tocopherol is a constituent of wheat-germ oil. Concentrates with high activity can be obtained from hempseed oil, cotton-seed oil, crude olive oil, green leafy vegetables, many seeds and muscle tissues. Viscous oil. Sol. most solvents. Shows absorption maxima at 292 m $\mu$  and 298 m $\mu$ . Thermostable. Resistant to light, acids, alkalis and mild oxidation.

*Allophanate*: cryst. from  $\text{MeOH}$ . M.p.  $158-60^\circ$ .

*p*-Nitrophenylurethane: cryst. from  $\text{MeOH}$ . M.p.  $129-31^\circ$ .

Evans, Emerson, Emerson, *J. Biol. Chem.*, 1936, **113**, 319.

*p*-Tolamidine (*p*-Tolonylamidine)

$\text{C}_8\text{H}_{10}\text{N}_2$  MW, 134

Plates from  $\text{C}_6\text{H}_6$ . M.p.  $101-2^\circ$ .

*B.HCl*: prisms +  $\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $213^\circ$ .

$\text{B}_2\text{H}_2\text{SO}_4$ : prisms +  $2\text{H}_2\text{O}$ . Does not melt below  $240^\circ$ .

$\text{B}_2\text{H}_2\text{SO}_4$ : plates from  $\text{EtOH}-\text{Et}_2\text{O}$ . M.p.  $240-1^\circ$ .

$\text{B.HNO}_2$ : needles. M.p.  $133^\circ$ .

$\text{B.HNO}_3$ : prisms from  $\text{EtOH}$ , m.p.  $154^\circ$ ; cryst. +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ , m.p.  $95^\circ$ .

$\text{B}_2\text{H}_2\text{PtCl}_6$ : yellow needles. M.p.  $225^\circ$ .

$\alpha$  :  $\beta$ -N-Di-Me :  $\text{C}_{10}\text{H}_{14}\text{N}_2$ . MW, 162. *B.HCl*: needles from  $\text{H}_2\text{O}$ . M.p.  $200^\circ$ .  $\text{B}_2\text{H}_2\text{PtCl}_6$ : cryst. +  $2\text{H}_2\text{O}$ . M.p.  $95^\circ$ .

$\beta$ -N-Et :  $\text{C}_{10}\text{H}_{14}\text{N}_2$ . MW, 162. *B.HCl*: needles. M.p.  $212^\circ$ .  $\text{B}_2\text{H}_2\text{PtCl}_6$ : needles +  $4\text{H}_2\text{O}$ . M.p.  $65^\circ$ .

$\beta$ -N-Phenyl :  $\text{C}_{14}\text{H}_{14}\text{N}_2$ . MW, 210. Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $149^\circ$ . Very sol.  $\text{EtOH}$ . Spar. sol.  $\text{C}_6\text{H}_6$ , hot ligroin.

$\beta$ -N-Acetyl : plates from  $\text{EtOH}-\text{AcOEt}$ . M.p.  $108^\circ$ .

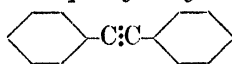
$\beta$ -N-*p*-Toluyyl : needles from  $\text{EtOH}$ . M.p.  $145^\circ$ .

Bernton, *Chem. Zentr.*, 1919, III, 329.

Glock, *Ber.*, 1888, **21**, 2653.

## Tolamine.

See Chloramine-T.

**Tolane** (sym.-*Diphenylacetylene*) $C_{14}H_{10}$ 

MW, 178

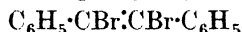
Plates or columns from EtOH. M.p. 62-5°. Dist. without decomp. Very sol. Et<sub>2</sub>O, hot EtOH. Spar. sol. cold EtOH. Heat of comb.  $C_p$  1738.2 Cal.,  $C_v$  1736.7 Cal. Latent heat of fusion 28.7 cal./gm. Ox.  $\rightarrow$  benzoic acid. Conc. H<sub>2</sub>SO<sub>4</sub> at 60°  $\rightarrow$  deoxybenzoin. Red.  $\rightarrow$  isostilbene + stilbene + dibenzyl.

$C_{14}H_{10} \cdot C_6H_5(NO_2)_3 \cdot 1 : 3 : 5$ : yellow plates. M.p. 96°.

*Picrate*: yellow plates. M.p. 111°.

Weissberger, *J. Chem. Soc.*, 1935, 855.

Fittig, *Ann.*, 1873, **168**, 74.

**Tolane dibromide** ( $\alpha$ :  $\beta$ -*Dibromostilbene*, 1 : 2-*dibromo*-1 : 2-*diphenylethylene*) $C_{14}H_{10}Br_2$ 

MW, 338

Exists in two forms, probably stereoisomeric.

 $\alpha$ -.

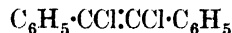
Plates or needles. M.p. 205-6°. Spar. sol. Et<sub>2</sub>O, hot EtOH. Difficultly volatile in steam. Heat with H<sub>2</sub>O at 170-80°  $\rightarrow$   $\beta$ -form. Hot alc. KOH  $\rightarrow$  tolane.

 $\beta$ -.

Needles. M.p. 64°. Very sol. Et<sub>2</sub>O, EtOH. Difficultly volatile in steam. Heat with H<sub>2</sub>O at 170-80°  $\rightarrow$   $\alpha$ -form. Hot alc. KOH  $\rightarrow$  tolane.

Staudinger, *Ber.*, 1916, **49**, 1972.

Limpricht, Schwanert, *Ber.*, 1871, **4**, 379.

**Tolane dichloride** ( $\alpha$ :  $\beta$ -*Dichlorostilbene*, 1 : 2-*dichloro*-1 : 2-*diphenylethylene*) $C_{14}H_{10}Cl_2$ 

MW, 249

Exists in two forms, probably stereoisomeric.

 $\alpha$ -.

Plates from EtOH. M.p. 140-2° (153°). B.p. 183°/18 mm. Sol. 130 parts EtOH at 24°. Very sol. Et<sub>2</sub>O. Heat.  $\rightarrow$   $\beta$ -form. HI(+P)  $\rightarrow$  dibenzyl. NaHg or alc. KOH  $\rightarrow$  tolane.

 $\beta$ -.

Needles. M.p. 63°. B.p. 178°/18 mm. Sol. 10 parts EtOH at 24°. Very sol. Et<sub>2</sub>O. Heat.  $\rightarrow$   $\alpha$ -form.

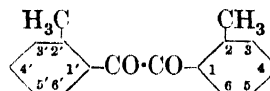
Staudinger, *Ber.*, 1916, **49**, 1971.

Loeb, *Ber.*, 1903, **36**, 3060.

See also last reference above.

**Tolidine.**

See Diaminodimethyldiphenyl.

**m-Tolil** (*Di-m-tolyl diketone*, 3 : 3'-*dimethylbenzil*) $C_{16}H_{14}O_2$ 

MW, 238

Yellowish-white needles from EtOH. M.p. 103°.

Ekecrantz, Ahlquist, *Chem. Zentr.*, 1908, II, 1689.

**p-Tolil** (*Di-p-tolyl diketone*, 4 : 4'-*dimethylbenzil*).

Yellow plates from EtOH. M.p. 104-5°. Sol. Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>. Alc. sol. + KOH  $\rightarrow$  violet col.

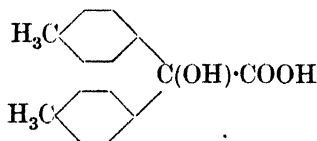
*Dioxime*: *syn*-.  $\alpha$ -*p*-tolildioxime. Plates or needles. M.p. 217°. Spar. sol. EtOH, Et<sub>2</sub>O, AcOH. *Diacetyl*: prisms. M.p. 133-4°. *Anti*-.  $\beta$ -*p*-tolildioxime. Needles. M.p. 225°. Very sol. EtOH. *Diacetyl*: m.p. 144°. *Amphi*-.  $\gamma$ -*p*-tolildioxime. Needles + 1CHCl<sub>3</sub> from CHCl<sub>3</sub>. Sinters about 200°. M.p. 229-32°. Long heat. at 200°  $\rightarrow$   $\beta$ -dioxime.

*Monohydrazone*: needles from EtOH. M.p. 139-40°.

*Dihydrazone*: needles from EtOH. M.p. 137°.

Vorländer, *Ber.*, 1911, **44**, 2461.

Tschugajew, Spiro, *Ber.*, 1908, **41**, 2221.

**p-Tolilic Acid** (4 : 4'-*Dimethylbenzilic acid*, *di-p-tolylglycollic acid*,  $\alpha$ -*hydroxy-di-p-tolylacetic acid*) $C_{16}H_{16}O_3$ 

MW, 256

Colourless needles from H<sub>2</sub>O. M.p. 135°. Sol. most org. solvents. Spar. sol. ligroin. Insol. cold H<sub>2</sub>O. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  purple col.

*Me ester*:  $C_{17}H_{18}O_3$ . MW, 270. Needles from EtOH. M.p. 82°. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

*Anhydride*:  $C_{32}H_{30}O_5$ . MW, 494. Prisms from EtOH. M.p. 164°.

*Acetyl*: cryst. from pet. ether. M.p. 92°.

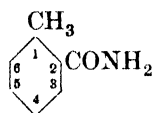
Gattermann, *Ann.*, 1906, **347**, 364.

Gisiger, *Ber.*, 1906, **39**, 3589.

**Toliminazole.**

See Methylbenziminazole.

## o-Toluamide

 $C_8H_9ON$ 

MW, 135

Plates from  $H_2O$ , needles from EtOH. M.p.  $147^\circ$  ( $138^\circ$ ). Sol. hot  $H_2O$ , EtOH. Spar. sol.  $Et_2O$ ,  $C_6H_6$ . NaHg in slightly acid sol.  $\rightarrow$  o-tolylcarbinol. NaHg in slightly alk. sol.  $\rightarrow$  dihydro-o-toluamide.  $EtOH + H_2SO_4 \rightarrow$  ethyl o-toluate.

N-Me:  $C_9H_{11}ON$ . MW, 149. Needles from  $H_2O$ . M.p.  $75^\circ$ .

N-Di-Me:  $C_{10}H_{13}ON$ . MW, 163. Liq. B.p.  $147^\circ/18$  mm.  $D^{25}_D$  1.033.

N-Benzoyl: needles from EtOH.Aq. M.p.  $158-9^\circ$ .

Noller, *Organic Syntheses*, 1933, XIII, 94.

## m-Toluamide.

Needles from  $Et_2O$ . M.p.  $97^\circ$  ( $93^\circ$ ). Spar. sol.  $Et_2O$ ,  $C_6H_6$ .

N-Me: m.p.  $44.5-45^\circ$ . Sol.  $H_2O$ , EtOH,  $Et_2O$ .

N-Di-Me: b.p.  $148^\circ/12$  mm.  $D^{15}_D$  1.043.

Remsen, Reid, *Am. Chem. J.*, 1899, **21**, 289.

## p-Toluamide.

Needles or plates from  $H_2O$ , needles from  $C_6H_6$ . M.p.  $155^\circ$  ( $165^\circ$ ). Very sol. hot  $H_2O$ , EtOH,  $Et_2O$ . Spar. sol. cold  $H_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

N-Me: plates from  $H_2O$ . M.p.  $145-145.5^\circ$ .

N-Di-Me: cryst. from EtOH. M.p.  $41^\circ$ . B.p.  $156^\circ/10$  mm. Sol.  $H_2O$ , EtOH.

N-Et:  $C_{10}H_{13}ON$ . MW, 163. Needles from  $H_2O$ . M.p.  $90^\circ$ .

N-Acetyl: prisms from EtOH.Aq. M.p.  $147^\circ$ .

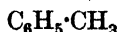
N-Benzoyl: needles from EtOH.Aq. M.p.  $119^\circ$ .

McMaster, Langreck, *J. Am. Chem. Soc.*, 1917, **39**, 106.

## Tolubenzyl cyanide.

See under Tolyacetic Acid.

## Toluene (Methylbenzene)

 $C_7H_8$ 

MW, 92

Constituent of many mineral oils. Important constituent of coal tar oil. Liq. F.p.  $-95.0^\circ$ . B.p.  $110.6^\circ$ ,  $102.6^\circ/600$  mm.,  $79.7^\circ/288$  mm.,  $14.5^\circ/14.56$  mm. Vap. press. at  $0^\circ$   $6.5$  mm., at  $20^\circ$   $22$  mm., at  $50^\circ$   $93.5$  mm., at  $60^\circ$   $141.5$  mm., at  $70^\circ$   $203$  mm., at  $80^\circ$   $292.5$  mm., at  $100^\circ$   $558$  mm. Very slightly sol.  $H_2O$ .  $D^{20}_4$  0.8845,

$D^{20}_4$  0.866.  $n^{20}_D$  1.49613. Heat of comb.  $C_p$   $937.4$  Cal.,  $C_v$   $938.5$  Cal.,  $C_p$  (vapour)  $955.68$  Cal. Crit. temp.  $320.8^\circ$ . Crit. press.  $41.6$  atm. Specific heat  $0.404$  cal./gm. at  $25^\circ$ . Latent heat of fusion  $0.0016$  Cal. Latent heat of vap.  $0.00094$  Cal. at  $110.2^\circ$ . Forms constant b.p. mixture with acetic acid, b.p.  $104-104.2^\circ$ .  
Picrate: pale yellow cryst. M.p.  $88.2^\circ$ .

Perkin, *J. Chem. Soc.*, 1896, **69**, 1191.

## Tolueneazoanisole.

See under Hydroxymethylazobenzene.

## Tolueneazobenzoic Acid.

See Methylazobenzene-carboxylic Acid.

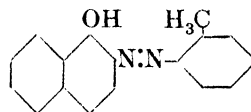
## Tolueneazocresol.

See Hydroxydimethylazobenzene.

5-p-Tolueneazo- $\psi$ -cumene.

See 2 : 4 : 5 : 4'-Tetramethylazobenzene.

**2-o-Tolueneazo-1-naphthol** (1 : 2-Naphthoquinone-2-o-tolylhydrazone)

 $C_{17}H_{14}ON_2$ 

MW, 262

Red leaflets. M.p.  $156^\circ$ . Sol. most org. solvents.

Me ether:  $C_{18}H_{16}ON_2$ . MW, 276. Orange-yellow needles from EtOH. M.p.  $84-5^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Less sol. ligroin. Conc.  $H_2SO_4 \rightarrow$  red sol.

Et ether:  $C_{19}H_{18}ON_2$ . MW, 290. Yellow needles from EtOH. M.p.  $51^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Less sol. pet. ether. Conc.  $H_2SO_4 \rightarrow$  ruby-red sol.

Zincke, Rathgen, *Ber.*, 1886, **19**, 2492.

Charrier, Ferreri, *Gazz. chim. ital.*, 1914, **44**, ii, 234.

**2-m-Tolueneazo-1-naphthol** (1 : 2-Naphthoquinone-2-m-tolylhydrazone).

Red needles from EtOH. M.p.  $117-18^\circ$ . Very sol.  $C_6H_6$ ,  $CHCl_3$ . Sol.  $Et_2O$ . Spar. sol. pet. ether. Insol. cold EtOH. Conc.  $H_2SO_4 \rightarrow$  red sol.

Me ether: yellow leaflets from EtOH. M.p.  $49-50^\circ$ . Sol. most org. solvents. Conc.  $H_2SO_4 \rightarrow$  red sol.

Et ether: orange-red prisms from EtOH. M.p.  $22^\circ$ . Very sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Sol. EtOH. Conc.  $H_2SO_4 \rightarrow$  ruby-red sol.

Charrier, Ferreri, *Gazz. chim. ital.*, 1914, **44**, ii, 235.

**2-*p*-Tolueneazo-1-naphthol** (1 : 2-*Naphthoquinone-2-p-tolylhydrazone*).

Red needles from EtOH. M.p. 145°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH. Spar. sol. pet. ether. Insol. alkalis. Conc. H<sub>2</sub>SO<sub>4</sub> → red sol.

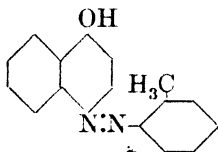
*Me ether*: orange-red needles from EtOH. M.p. 77–8°. Sol. Et<sub>2</sub>O, boiling EtOH, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. pet. ether. Conc. H<sub>2</sub>SO<sub>4</sub> → ruby-red sol.

*Et ether*: orange-yellow needles from EtOH. M.p. 51°. Sol. most org. solvents. Conc. H<sub>2</sub>SO<sub>4</sub> → ruby-red sol.

See previous reference and also

Charrier, Casale, *Gazz. chim. ital.*, 1914, **44**, i, 611.

**4-*o*-Tolueneazo-1-naphthol** (1 : 4-*Naphthoquinone-o-tolylhydrazone*)



C<sub>17</sub>H<sub>14</sub>ON<sub>2</sub>

MW, 262

Red needles from 66% EtOH. M.p. 144–6°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH. Less sol. pet. ether.

*Me ether*: C<sub>18</sub>H<sub>16</sub>ON<sub>2</sub>. MW, 276. Reddish-brown needles. M.p. 93°. Sol. usual org. solvents.

*Et ether*: C<sub>19</sub>H<sub>18</sub>ON<sub>2</sub>. MW, 290. Red plates from EtOH. M.p. 94°.

Zincke, Rathgen, *Ber.*, 1886, **19**, 2488.

**4-*p*-Tolueneazo-1-naphthol** (1 : 4-*Naphthoquinone-p-tolylhydrazone*).

Dark red cryst. from PhNO<sub>2</sub>. M.p. 209–10°. Sol. Me<sub>2</sub>CO, PhNO<sub>2</sub>, aniline. Spar. sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH.

*Me ether*: cryst. M.p. 103–4°.

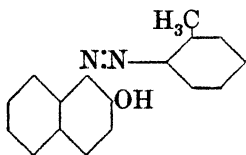
*Et ether*: red needles from EtOH. M.p. 126–7°.

*Acetyl*: yellowish needles from pet. ether. M.p. 101–2°.

See previous reference and also

Witt, Schmidt, *Ber.*, 1892, **25**, 1019.

**1-*o*-Tolueneazo-2-naphthol** (1 : 2-*Naphthoquinone-1-o-tolylhydrazone*)



C<sub>17</sub>H<sub>14</sub>ON<sub>2</sub>

MW, 262

Red needles from AcOH. M.p. 131° (130°).

*Me ether*: C<sub>18</sub>H<sub>16</sub>ON<sub>2</sub>. MW, 276. Red plates from EtOH. M.p. 58°. Sol. most org. solvents. Conc. H<sub>2</sub>SO<sub>4</sub> → red sol. *B,2HNO<sub>3</sub>*: green needles. M.p. 71° decomp. Sol. EtOH, Et<sub>2</sub>O. Mod. sol. CHCl<sub>3</sub>. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin.

*Et ether*: C<sub>19</sub>H<sub>18</sub>ON<sub>2</sub>. MW, 290. Red needles from EtOH. M.p. 36°. Sol. most org. solvents. *B,2HNO<sub>3</sub>*: green leaflets. M.p. 62–3°. Mod. sol. EtOH, CHCl<sub>3</sub>. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin.

Norman, *J. Chem. Soc.*, 1912, **101**, 1920.

Charrier, Ferreri, *Gazz. chim. ital.*, 1912, **42**, ii, 124; 1913, **43**, i, 549.

**1-*m*-Tolueneazo-2-naphthol** (1 : 2-*Naphthoquinone-1-m-tolylhydrazone*).

Red needles from C<sub>6</sub>H<sub>6</sub>. M.p. 141° (137°). Sol. most org. solvents. Conc. H<sub>2</sub>SO<sub>4</sub> → violet sol.

*Me ether*: red plates from EtOH. M.p. 81°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Spar. sol. pet. ether. Conc. acids → red sols. *B,2HNO<sub>3</sub>*: green leaflets. M.p. 72°. Sol. EtOH, CHCl<sub>3</sub>. Less sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether.

*Et ether*: red leaflets from ligroin. M.p. 84°. Sol. most org. solvents. *B,2HNO<sub>3</sub>*: green leaflets. M.p. 84° decomp. Sol. EtOH, CHCl<sub>3</sub>. Insol. C<sub>6</sub>H<sub>6</sub>.

See previous references.

**1-*p*-Tolueneazo-2-naphthol** (1 : 2-*Naphthoquinone-1-p-tolylhydrazone*).

Red needles or plates from AcOH. M.p. 134–5°. Sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, AcOH.

*Me ether*: red plates from EtOH. M.p. 68°. Sol. most org. solvents. Conc. H<sub>2</sub>SO<sub>4</sub> → ruby-red sol. *B,2HNO<sub>3</sub>*: red needles. M.p. 77° decomp. Sol. warm CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>.

*Et ether*: red needles from EtOH. M.p. 51°. Very sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Less sol. EtOH, pet. ether. *B,2HNO<sub>3</sub>*: red leaflets. M.p. 94° decomp. Mod. sol. EtOH, CHCl<sub>3</sub>. Less sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. ligroin.

See previous references.

**Tolueneazophenetole.**

See under Hydroxy-methylazobenzene.

**Tolueneazophenol.**

See Hydroxy-methylazobenzene.

**Tolueneazotoluidine.**

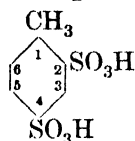
See Aminodimethylazobenzene.

**Tolueneazoxylenol.**

See 2-Hydroxy-3 : 5 : 4'-trimethylazobenzene.

**Toluene-dicarboxylic Acid.**

See Methylphthalic Acid, Methylisophthalic Acid, and Methylterephthalic Acid.

**Toluene-2 : 4-disulphonic Acid**

$C_7H_8O_6S_2$  MW, 252

Thick oil. Ox. of K salt  $\rightarrow$  2 : 4-disulphobenzoic acid. Fuse K salt with  $H\cdot COONa \rightarrow$  4-methylsulphthalic acid.

*Difluoride* :  $C_7H_6O_4F_2S_2$ . MW, 256. Plates from petrol. M.p.  $87-8^\circ$ . Sol.  $Me_2CO$ ,  $AcOEt$ ,  $PhNO_2$ . Mod. sol.  $EtOH$ ,  $Et_2O$ ,  $CS_2$ . Spar. sol. pet. ether.

*Dichloride* :  $C_7H_6O_4Cl_2S_2$ . MW, 289. Prisms from  $Et_2O$ . M.p.  $56^\circ$ . Very sol.  $Et_2O$ ,  $C_6H_6$ .

*Dibromide* :  $C_7H_6O_4Br_2S_2$ . MW, 378. Cryst. from  $Et_2O$ . M.p.  $78^\circ$ .

*Diamide* :  $C_7H_{10}O_4N_2S_2$ . MW, 250. Prisms from  $EtOH$  or  $H_2O$ . M.p.  $190-1^\circ$ . Sol. warm  $H_2O$ . Very sol.  $EtOH$ ,  $NH_4OH$ .

*Dianilide* : prisms from  $EtOH.Aq.$ , needles from  $C_6H_6$ , plates from  $Me_2CO$ . M.p.  $189^\circ$ .

*Di-o-toluidide* : cryst. M.p.  $170-1^\circ$ .

*Di-m-toluidide* : cryst. M.p.  $138^\circ$ .

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 38.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 757.

Gnehm, Forrer, *Ber.*, 1877, **10**, 542.

**Toluene-2 : 5-disulphonic Acid.**

*Dichloride* : prisms or plates from  $C_6H_6$  or  $C_6H_6$ -pet. ether. M.p.  $98^\circ$ . Very sol.  $AcOEt$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol. pet. ether.

*Diamide* : prisms. M.p.  $224^\circ$ . Spar. sol.  $H_2O$ .

*Dianilide* : needles from  $C_6H_6$  or  $EtOH.Aq.$  M.p.  $178^\circ$ .

See second reference above.

**Toluene-2 : 6-disulphonic Acid.**

*Dichloride* : prisms from pet. ether or  $C_6H_6$ . M.p.  $88^\circ$ . Very sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. pet. ether.

*Diamide* : needles. Does not melt below  $260^\circ$ . Mod. sol.  $EtOH$ . Spar. sol.  $H_2O$ .

*Dianilide* : prisms from  $EtOH.Aq.$  or  $C_6H_6$ . M.p.  $162^\circ$ .

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 771.

**Toluene-3 : 4-disulphonic Acid.**

Forms cryst. spar. sol. Ba salt.

*Dichloride* : scales from  $C_6H_6$ -pet. ether, m.p.  $111^\circ$ ; cryst. +  $\frac{1}{2}C_6H_6$  from  $C_6H_6$ , m.p.  $70-80^\circ$ .

*Diamide* : m.p.  $235-9^\circ$  (slight decomp.). Very sol.  $H_2O$ ,  $EtOH$ .

*Dianilide* : plates from  $Me_2CO$ , prisms from  $EtOH$ . M.p.  $190^\circ$ .

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 751.

Klason, *Ber.*, 1887, **20**, 356.

**Toluene-3 : 5-disulphonic Acid.**

Needles. Very sol.  $H_2O$ ,  $EtOH$ .

*Dichloride* : prisms from  $Et_2O$ . M.p.  $95^\circ$ .

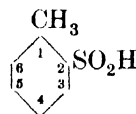
Very sol.  $Et_2O$ . Spar. sol. pet. ether.

*Diamide* : prisms from  $H_2O$ . M.p.  $216^\circ$ . Sol. hot  $H_2O$ ,  $EtOH$ . Spar. sol. cold  $H_2O$ .

*Dianilide* : scales from  $C_6H_6$  or  $EtOH.Aq.$  M.p.  $153^\circ$ .

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 734, 738.

Richter, *Ann.*, 1885, **230**, 326.

**Toluene-o-sulphinic Acid**

$C_7H_8O_2S$  MW, 156

Needles. M.p.  $80^\circ$ . Very sol. most org. solvents. Easily decomp. Salts very sol.  $H_2O$ .

Höchst, D.R.P., 224,019, (*Chem. Zentr.*, 1910, II, 513).

Gattermann, *Ber.*, 1899, **32**, 1140.

**Toluene-m-sulphinic Acid.**

Unstable oil with strong bleaching properties.

Troeger, Hille, *J. prakt. Chem.*, 1905, **71**, 207.

**Toluene-p-sulphinic Acid.**

*d*l-.

*Et ester* :  $C_9H_{12}O_2S$ . MW, 184. B.p.  $60-1^\circ$ /high vacuum.  $n_D^{25}$  1.5309.  $[\alpha]_{5461}^{25} + 0.92^\circ$ .

*l*-.

*Et ester* : b.p.  $63^\circ$ /high vacuum.  $n_D^{25}$  1.5309.  $[\alpha]_{5461}^{25} - 6.72^\circ$ .

*Butyl ester* : b.p.  $84^\circ$ /less than 0.1 mm.  $D_4^{25}$  1.066.  $n_D^{25}$  1.5195.

*Anilide* : plates from  $EtOH.Aq.$  M.p.  $134^\circ$ . B.p.  $75-6^\circ$ /less than 0.1 mm.  $[\alpha]_{5461}^{17} - 1.1^\circ$  in  $CHCl_3$ .

*dl*-.

Dark plates or needles from  $H_2O$ . M.p.  $85^\circ$ . Very sol.  $EtOH$ ,  $Et_2O$ . Spar. sol. cold  $H_2O$ , hot  $C_6H_6$ . Hygroscopic. Very readily oxidised to toluene-*p*-sulphonic acid.  $Zn + H_2SO_4 \rightarrow$  *p*-thiocresol. Alc. MeI  $\rightarrow$  methyl *p*-tolyl sulphone. Forms spar. sol. Ag salt.

$NH_4$  salt : needles from  $EtOH$ . M.p.  $175^\circ$  decomp.

*Et ester*: b.p. 99–104°/0.1 mm.  $n_D^{25}$  1.5309.  $D_4^{25}$  1.114.

*Butyl ester*: b.p. 90–95°/0.1 mm.  $D_4^{25}$  1.066.  $n_D^{25}$  1.5195.

*Anhydride*:  $C_{14}H_{14}O_3S_2$ . MW, 294. M.p. 75°. Decomp. on standing.

*Chloride*:  $C_7H_7OClS$ . MW, 174.5. Needles. M.p. 54–8°.

*Amide*:  $C_7H_7ONS$ . MW, 155. Needles. M.p. 120°.

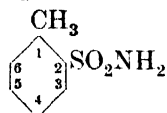
*Anilide*: m.p. 138°.

Whitmore, Hamilton, *Organic Syntheses*, Collective Vol. I, 479.

Phillips, *J. Chem. Soc.*, 1925, 2552.

v. Braun, Kaiser, *Ber.*, 1923, 56, 549.

### Toluene-*o*-sulphonamide



$C_7H_7O_2NS$

MW, 171

Octahedra from EtOH, prisms from  $H_2O$ . M.p. 156–3°. Sol. 958 parts  $H_2O$  at 9°, 28 parts EtOH at 5°. Electrolytic oxidation of alk. metal salts  $\rightarrow$  saccharin.

*N-Me*:  $C_8H_{11}O_2NS$ . MW, 185. Plates from  $C_6H_6$ -ligroin. M.p. 74–5°. Very sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ . Spar. sol. ligroin, hot  $H_2O$ . Insol. cold  $C_6H_6$ .

*N-Chloro deriv.*:  $C_7H_7O_2NClS$ . MW, 205.5. *Na salt*: prisms +  $2H_2O$ . Explodes (anhyd.) at 170–5°. *K salt*: prisms +  $H_2O$ . Explodes (anhyd.) about 145°.

*N-Dichloro deriv.*:  $C_7H_7O_2NCl_2S$ . MW, 240. Plates from  $CHCl_3$ -pet. ether. M.p. 33°.

*N-Bromo deriv.*:  $C_7H_7O_2NBrS$ . MW, 250. *Na salt*: yellow plates +  $1H_2O$ . Explodes (anhyd.) about 135–40°. *K salt*: yellow plates. Decomp. (anhyd.) at 130–5°.

*N-Dibromo deriv.*:  $C_7H_7O_2NBr_2S$ . MW, 329. Orange plates from  $CHCl_3$ -pet. ether. M.p. 80°. Very sol.  $CHCl_3$ . Spar. sol. pet. ether.

*N-Benzoyl*: *N*-*o*-toluenesulphonylbenzamide. Plates or prisms from  $Et_2O$ . M.p. 110–12°.

Ullmann, *Encyclopedia of Tech. Chem.*, (Edition 2. Berlin-Vienna 1928), II, 251.

Chattaway, *J. Chem. Soc.*, 1905, 87, 152.

### Toluene-*m*-sulphonamide.

Plates or cryst. aggregates from  $H_2O$ , prisms from EtOH. M.p. 108°. Sol. 130 parts  $H_2O$  at 25°. Sol. EtOH.

Griffin, *Am. Chem. J.*, 1897, 19, 174.

### Toluene-*p*-sulphonamide.

Plates +  $2H_2O$  from  $H_2O$ . M.p. 105°, anhyd. 137.5°. Sol. 515 parts  $H_2O$  at 9°, 13.5 parts EtOH at 5°.

*N-Me*:  $C_8H_{11}O_2NS$ . MW, 185. Plates from EtOH.Aq. M.p. 78–9°.

*N-Di-Me*:  $C_9H_{13}O_2NS$ . MW, 199. Needles from petrol. M.p. 86–7°. Sol. AcOEt,  $C_6H_6$ . Mod. sol.  $Et_2O$ , EtOH. Spar. sol.  $H_2O$ .

*N-Et*:  $C_9H_{13}O_2NS$ . MW, 199. Cryst. from ligroin, plates from EtOH.Aq. M.p. 64°.

*N-Di-Et*:  $C_{11}H_{17}O_2NS$ . MW, 227. Cryst. from ligroin. M.p. 60°. Spar. sol.  $H_2O$ , ligroin.

*N-Propyl*:  $C_{10}H_{15}O_2NS$ . MW, 213. Cryst. from ligroin. M.p. 52°.

*N-Butyl*:  $C_{11}H_{17}O_2NS$ . MW, 227. M.p. 43°.

*N-Hexyl*:  $C_{13}H_{21}O_2NS$ . MW, 255. M.p. 62°.

*N-Heptyl*:  $C_{14}H_{23}O_2NS$ . MW, 269. M.p. 27°.

*N-Benzyl*:  $C_{14}H_{15}O_2NS$ . MW, 261. Prisms from EtOH. M.p. 115–16°.

*N-Dibenzyl*:  $C_{21}H_{21}O_2NS$ . MW, 351. Needles from MeOH. M.p. 80.8°.

*N-Chloro*: *Na salt*: see Chloramine-T. *K salt*: plates +  $H_2O$ . Explodes (anhyd.) about 160–5°.

*N-Dichloro*: see Dichloramine-T.

*N-Bromo*:  $C_7H_8O_2NBrS$ . MW, 250. *Na salt*: yellow prisms +  $3H_2O$ . Decomp. (anhyd.) at 145–50°. *K salt*: yellow plates +  $2H_2O$ . Decomp. (anhyd.) at 145–50°.

*N-Dibromo*:  $C_7H_7O_2NBr_2S$ . MW, 329. Orange plates from  $CHCl_3$ . M.p. 104°.

*N-Acetyl*: needles. M.p. 139°.

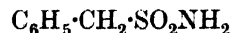
*N-Benzoyl*: prisms or needles from EtOH. M.p. 147–50°.

Steinkopf, *J. prakt. Chem.*, 1927, 117, 25.

Chattaway, *J. Chem. Soc.*, 1905, 87, 152.

Wolkow, *Z. Chem.*, 1870, 323, 578.

### Toluene- $\alpha$ -sulphonamide (Benzylsulphonamide)



$C_7H_9O_2NS$

MW, 171

Prisms or needles from hot  $H_2O$ , needles from EtOH. M.p. 105°. Very sol. hot  $H_2O$ , EtOH.

*N-Me*:  $C_8H_{11}O_2NS$ . MW, 185. Needles. M.p. 109–10°. Sol. hot  $H_2O$ , EtOH,  $Et_2O$ .

*N-Di-Me*:  $C_9H_{13}O_2NS$ . MW, 199. Plates from  $CHCl_3$ -ligroin. M.p. 101°.

*N-Et*:  $C_9H_{13}O_2NS$ . MW, 199. Leaflets from  $Et_2O$ -ligroin. M.p. 65–6°.

N-Di-Et:  $C_{11}H_{17}O_2NS$ . MW, 227. Leaflets from  $Et_2O$ -ligroin. M.p. 29°.

Ingold, Ingold, Shaw, *J. Chem. Soc.*, 1927, 818.

Curtius, Haas, *J. prakt. Chem.*, 1921, 102, 102, 104, 106.

Johnson, Ambler, *J. Am. Chem. Soc.*, 1914, 36, 381.

### Toluenesulphonbromide.

See under Toluenesulphonic Acid.

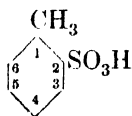
### Toluenesulphonchloride.

See under Toluenesulphonic Acid.

### Toluenesulphonfluoride.

See under Toluenesulphonic Acid.

### Toluene-*o*-sulphonic Acid



$C_7H_8O_3S$

MW, 172

Hygroscopic plates +  $2H_2O$ . Heat. at 140–50° → toluene-*p*-sulphonic acid. Ox. → *p*-sulphobenzoic acid. KOH fusion → *o*-cresol + salicylic acid. Gives spar. sol. Ag, Pb salts.

-Menthyl ester:  $C_{17}H_{26}O_3S$ . MW, 310. Prisms. M.p. 78°.

Phenylester:  $C_{13}H_{12}O_3S$ . MW, 248. M.p. 52°.

*o*-Nitrophenyl ester:  $C_{13}H_{11}O_5NS$ . MW, 293. Plates from EtOH. M.p. 131–4°.

*o*-Tolyl ester:  $C_{14}H_{14}O_3S$ . MW, 262. Cryst. from EtOH. M.p. 50–1°.

*m*-Tolyl ester: cryst. from EtOH. M.p. 60°.

*p*-Tolyl ester: cryst. from EtOH. M.p. 70–1°.

Fluoride: toluene-*o*-sulphonfluoride.  $C_7H_7O_2FS$ . MW, 174. Oil. B.p. 223–5°, 146·2°/83 mm., 133·9°/56·5 mm.  $n_D^{20}$  1·5007.

Chloride: toluene-*o*-sulphonchloride.  $C_7H_7O_2ClS$ . MW, 190·5. Cryst. from  $CHCl_3$ . M.p. 67·5°. B.p. 126°/10 mm.

Bromide: toluene-*o*-sulphonbromide.  $C_7H_7O_2BrS$ . MW, 235. Cryst. M.p. 13°. B.p. 137·5–138°/10 mm.

Amide: see Toluene-*o*-sulphonamide.

Anilide: plates from EtOH.Aq. M.p. 136°.

*o*-Toluidide: m.p. 134°.

Terlinck, *Chem. Abstracts*, 1927, 21, 1978.

Steinkopf, *J. prakt. Chem.*, 1927, 117, 38.

Holleman, Caland, *Ber.*, 1911, 44, 2505.

Ullmann, Lehner, *Ber.*, 1905, 38, 732.

### Toluene-*m*-sulphonic Acid.

Free acid is an oil.

Chloride: toluene-*m*-sulphonchloride. F.p. 11·7°. Decomp. by boiling  $H_2O$ .

Amide: see Toluene-*m*-sulphonamide.

Anilide: prisms from EtOH. M.p. 96°.

*o*-Toluidide: prisms and plates. M.p. 108°.

*m*-Toluidide: m.p. 103°.

*p*-Toluidide: prisms. M.p. 106°.

Holleman, Caland, *Ber.*, 1911, 44, 2504, 2515.

Griffin, *Am. Chem. J.*, 1897, 19, 173, 189.

### Toluene-*p*-sulphonic Acid.

Hygroscopic plates +  $1H_2O$  from  $H_2O$ . M.p. 92° (104–5°). Ox. → *p*-sulphobenzoic acid. KOH fusion → *p*-cresol + *p*-hydroxybenzoic acid.

Me ester:  $C_8H_{10}O_3S$ . MW, 186. Cryst. from  $Et_2O$ -ligroin. M.p. 28°. Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

Et ester:  $C_9H_{12}O_3S$ . MW, 200. Cryst. from AcOEt. M.p. 33–4°. B.p. 173°/15 mm.  $D_4^{25}$  1·166.

Propyl ester:  $C_{10}H_{14}O_3S$ . MW, 214. B.p. 154–6°/3 mm.  $D_4^{20}$  1·144.  $n_D^{20}$  1·4998.

Butyl ester:  $C_{11}H_{16}O_3S$ . MW, 228. B.p. 163–5°/3 mm.  $D_4^{20}$  1·120.  $n_D^{20}$  1·5050.

sec.-*n*-Butyl ester: decomp. on heating.  $D_4^{20}$  1·140.  $n_D^{20}$  1·5100.

Isobutyl ester: b.p. 163–5°/3 mm.  $D_4^{20}$  1·125.  $n_D^{20}$  1·5050.

*n*-Amyl ester:  $C_{12}H_{18}O_3S$ . MW, 242. B.p. 169–70°/3 mm.  $D_4^{20}$  1·140.  $n_D^{20}$  1·5100.

Phenyl ester:  $C_{13}H_{12}O_3S$ . MW, 248. Needles from EtOH. M.p. 95–6°. Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol.  $H_2O$ .

Benzyl ester:  $C_{14}H_{14}O_3S$ . MW, 262. Cryst. M.p. 58°.

*l*-Menthyl ester:  $C_{17}H_{26}O_3S$ . MW, 310. Needles. M.p. 97°.  $[\alpha]_D^{20}$  –66·8° in  $CHCl_3$ . Spar. sol. cold EtOH.

*1*-Naphthyl ester:  $C_{17}H_{14}O_3S$ . MW, 298. Needles from EtOH. M.p. 83–4°.

*2*-Naphthyl ester: plates from EtOH or  $C_6H_6$ -ligroin. M.p. 125°.

Anhydride:  $C_{14}H_{14}O_5S_2$ . MW, 326. Cryst. from  $Et_2O$ . M.p. 122–5°.

Fluoride: toluene-*p*-sulphonfluoride.  $C_7H_7O_2FS$ . MW, 174. Needles from pet. ether. M.p. 43–4°. B.p. 112·5°/16 mm.

Chloride: toluene-*p*-sulphonchloride.  $C_7H_7O_2ClS$ . MW, 190·5. Cryst. from  $Et_2O$  or pet. ether. M.p. 71°. B.p. 145–6°/15 mm.

Bromide: toluene-*p*-sulphonbromide.  $C_7H_7O_2BrS$ . MW, 235. Columns. M.p. 96°.

Iodide: toluene-*p*-sulphoniodide.  $C_7H_7O_2IS$ . MW, 282. Yellow powder. M.p. 84–5° decomp.

Amide: see Toluene-*p*-sulphonamide.

Hydrazide: plates or needles from  $H_2O$ . M.p. 112°.



*Phenylhydrazide*: needles from EtOH. M.p. 155° decomp.

*Azide*: plates. M.p. 22°.

*Anilide*: needles from Et<sub>2</sub>O-EtOH. M.p. 103°.

*m-Nitroanilide*: needles from EtOH.Aq. M.p. 139°.

*p-Nitroanilide*: yellow prisms from EtOH, needles from C<sub>6</sub>H<sub>6</sub>. M.p. 191°.

*o-Toluidide*: prisms from EtOH, needles from AcOH.Aq. M.p. 110°.

*m-Toluidide*: m.p. 114°.

*p-Toluidide*: needles from AcOH. M.p. 118-19°.

*1-Naphthylamide*: prisms from EtOH. M.p. 157°.

*2-Naphthylamide*: needles, plates or prisms from EtOH. M.p. 133°.

*A,CH<sub>3</sub>NH<sub>2</sub>*: hygroscopic cryst. M.p. 125°.

*A,(CH<sub>3</sub>)<sub>2</sub>NH*: prisms. M.p. 78°.

*A,(CH<sub>3</sub>)<sub>3</sub>N*: rosettes. M.p. 92°.

*A,C<sub>2</sub>H<sub>5</sub>NH<sub>2</sub>*: m.p. 111°.

*A,(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>NH*: clusters of cryst. M.p. 88°.

*A,(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>N*: m.p. 65°.

Curtius, Kraemer, *J. prakt. Chem.*, 1930, **125**, 323.

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 22.

Gilman, Beaber, *J. Am. Chem. Soc.*, 1925, **47**, 522.

Holleman, Caland, *Ber.*, 1911, **44**, 2505.

**Toluene- $\alpha$ -sulphonic Acid** (*Benzylsulphonic acid*)



C<sub>7</sub>H<sub>8</sub>O<sub>3</sub>S

MW, 172

Hygroscopic cryst. Forms spar. sol. Ba salt.

*Fluoride*: C<sub>7</sub>H<sub>7</sub>O<sub>2</sub>FS. MW, 174. Needles from petrol. M.p. 90-1°.

*Chloride*: C<sub>7</sub>H<sub>7</sub>O<sub>2</sub>ClS. MW, 190.5. Prisms from Et<sub>2</sub>O, needles from C<sub>6</sub>H<sub>6</sub>. M.p. 93°. Very sol. Et<sub>2</sub>O, warm C<sub>6</sub>H<sub>6</sub>.

*Amide*: see Toluene- $\alpha$ -sulphonamide.

*Hydrazide*: plates from EtOH. M.p. 131-2° decomp.

*Phenylhydrazide*: m.p. 173° decomp.

*Azide*: needles from EtOH. M.p. 54°.

*Anilide*: needles from EtOH. M.p. 102°.

*p-Nitroanilide*: cryst. from EtOH.Aq. M.p. 155°.

*o-Toluidide*: m.p. 83°.

*m-Toluidide*: m.p. 75°.

*p-Toluidide*: prisms from EtOH. M.p. 113°.

*1-Naphthylamide*: yellow needles from EtOH. M.p. 166°.

*2-Naphthylamide*: needles. M.p. 148.5°.

Medwedew, Alexejewa, *Ber.*, 1932, **65**, 131.

Clutterbuck, Cohen, *J. Chem. Soc.*, 1923, **123**, 2507.

Johnson, Ambler, *J. Am. Chem. Soc.*, 1914, **36**, 381.

Fromm, Palma, *Ber.*, 1906, **39**, 3312.

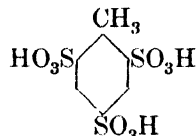
**Toluenesulphoniodide.**

See under Toluene-*p*-sulphonic Acid.

**Toluene-tricarboxylic Acid.**

See Methyltrimellitic Acid and Methyltrimesic Acid.

**Toluene-2 : 4 : 6-trisulphonic Acid**



C<sub>7</sub>H<sub>8</sub>O<sub>6</sub>S<sub>3</sub>

MW, 332

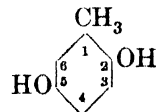
Needles + 6H<sub>2</sub>O (after drying in vacuo). Retains 3H<sub>2</sub>O at 100°. M.p. 145°. The trihydrate is hygroscopic. Very sol. H<sub>2</sub>O.

*Trichloride*: C<sub>7</sub>H<sub>5</sub>O<sub>6</sub>Cl<sub>3</sub>S<sub>3</sub>. MW, 387.5. Plates from CHCl<sub>3</sub>. M.p. 153°. Spar. sol. boiling Et<sub>2</sub>O.

*Triamide*: C<sub>7</sub>H<sub>11</sub>O<sub>6</sub>N<sub>3</sub>S<sub>3</sub>. MW, 329. Cryst. Does not melt below 300°. Sol. warm NH<sub>3</sub>. Insol. H<sub>2</sub>O.

Claesson, *Ber.*, 1881, **14**, 307.

**Toluhydroquinone** (*Toluquinol*, *homohydroquinone*, *homoquinol*, *2-methylhydroquinone*, *2 : 5-dihydroxytoluene*, *hydrotoluquinone*)



C<sub>7</sub>H<sub>8</sub>O<sub>2</sub>

MW, 124

Plates from C<sub>6</sub>H<sub>6</sub>. M.p. 124-5°. B.p. 163°/11 mm. Very sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>, ligroin. Sol. alkalis, alk. carbonates. Sublimes. Heat of comb. C<sub>7</sub> 836.3 Cal. Reduces NH<sub>3</sub>.AgNO<sub>3</sub> and Fehling's. CrO<sub>3</sub> → *p*-toluquinone. CaCl<sub>2</sub> → bluish-green → brown col. FeCl<sub>3</sub> → brownish-red or yellow col.

*2-Me ether*: C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>. MW, 138. Needles from C<sub>6</sub>H<sub>6</sub>-petrol. M.p. 46-46.5°. Very sol. cold EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether, cold H<sub>2</sub>O.

*5-Me ether*: needles from hot H<sub>2</sub>O. M.p. 70.5-71.5°. B.p. 240-5°. Very sol. cold Et<sub>2</sub>O, EtOH, CHCl<sub>3</sub>. Spar. sol. pet. ether, cold H<sub>2</sub>O.

*Di-Me ether*:  $C_9H_{12}O_2$ . MW, 152. Cryst. M.p. 15°. B.p. 214–18°. Volatile in steam.

*5-Et ether*:  $C_9H_{12}O_2$ . MW, 152. Prisms or needles from EtOH.Aq. M.p. 55–55.5°. Very sol. hot ligroin and most org. solvents. Spar. sol. hot  $H_2O$ . Volatile in steam.

*Di-Et ether*:  $C_{11}H_{16}O_2$ . MW, 180. Needles from cold ligroin. M.p. 24–5°. B.p. 247–9°.

*Acetyl deriv.*: needles from pet. ether. M.p. 92°.

*Diacetyl*: needles or prisms from  $H_2O$ , AcOH or ligroin. M.p. 49°.

*Dibenzoyl*: cryst. M.p. 119–20°.

Schmid, *Monatsh.*, 1911, **32**, 437.

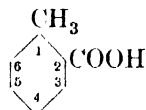
Bamberger, *Ann.*, 1912, **390**, 175.

Henderson, Boyd, *J. Chem. Soc.*, 1910, **97**, 1667.

### $\alpha$ -Toluic Acid.

See Phenylacetic Acid.

### *o*-Toluic Acid (2-Methylbenzoic acid)



$C_8H_8O_2$  MW, 136

Prisms from  $H_2O$ . M.p. 107–8° (102°). B.p. 258.5–259°. Very sol. EtOH. Sol. hot  $H_2O$ . Volatile in steam. Heat of comb.  $C_p$  929.8 Cal.,  $C_p$  929.4 Cal.  $k = 1.35 \times 10^{-4}$  at 25°. Ox.  $\rightarrow$  phthalic acid. Na + amyl alcohol  $\rightarrow$  *trans*-hexahydro-*o*-toluic acid.

*Na salt*: plates +  $2H_2O$ . M.p. 227–8°.

*K salt*: m.p. 188–9°.

*Me ester*:  $C_9H_{10}O_2$ . MW, 150. Cryst. below –50°. B.p. 213°, 97°/15 mm.  $D^{15}_D$  1.073.

*Et ester*:  $C_{10}H_{12}O_2$ . MW, 164. B.p. 227°, 102–102.5°/13 mm.  $D^{21.6}_D$  1.0325.  $n^{21.6}_D$  1.507.

*d-Amyl ester*:  $C_{13}H_{18}O_2$ . MW, 206. B.p. 265–8°.  $D^{20}_D$  0.985.  $n^{19.6}_D$  1.4984.  $[\alpha]^{20}_D + 5.94^\circ$ .

*l-Menthyl ester*:  $C_{18}H_{26}O_2$ . MW, 274. B.p. 191°/15 mm.  $D^{20}_D$  0.9982.  $[\alpha]^{20}_D - 84.35^\circ$ .

*Phenyl ester*:  $C_{14}H_{12}O_2$ . MW, 212. Yellow oil. B.p. 306°/754 mm.

*Benzyl ester*:  $C_{15}H_{14}O_2$ . MW, 226. Oil. B.p. 315°.  $D^{17}_D$  1.12.

*p-Nitrobenzyl ester*:  $C_{15}H_{13}O_4N$ . MW, 271. Cryst. from EtOH.Aq. M.p. 90.7°.

*p-Bromphenacyl ester*:  $C_{16}H_{13}O_3Br$ . MW, 333. M.p. 56.9°.

*Anhydride*:  $C_{16}H_{14}O_3$ . MW, 254. Cryst. from  $Et_2O$  or  $C_6H_6$ . M.p. 39°. B.p. 220–1°/11 mm.

*Chloride*:  $C_8H_7OCl$ . MW, 154.5. B.p. 206–8°, 110–11°/29 mm., 99–100°/14 mm.

*Bromide*:  $C_8H_7OBr$ . MW, 199. B.p. 133–6°/37 mm.

*Amide*: see *o*-Toluamide.

*Nitrile*: see *o*-Tolunitrile.

*Imide*: cryst. from EtOH. M.p. 147–8°.

*Hydrazide*: needles from EtOH.Aq. or  $Et_2O$ . M.p. 124°.

*Anilide*: m.p. 125°.

*p-Toluidide*: m.p. 144°.

Clarke, Taylor, *Organic Syntheses*, 1931, **XI**, 96.

### *m*-Toluic Acid (3-Methylbenzoic acid).

Prisms from  $H_2O$ . M.p. 111–13°. B.p. 263°. Sol. 1170 parts  $H_2O$  at 15°, 60 parts at 100°. Very sol. EtOH,  $Et_2O$ . Sublimes. Volatile in steam. Heat of comb.  $C_p$  928.5 Cal.,  $C_p$  929.1 Cal.  $k = 5.6 \times 10^{-5}$  at 25°. Ox.  $\rightarrow$  isophthalic acid. Red.  $\rightarrow$  hexahydro-*m*-toluic acid.

*Na salt*: m.p. about 310°.

*Me ester*: oil. B.p. 215°.  $D^{15}_D$  1.066.

*Et ester*: b.p. 227°, 133°/38 mm., 103–5°/10 mm.  $D^{20}_D$  1.0301.  $n^{21.6}_D$  1.505.

*Propyl ester*:  $C_{11}H_{14}O_2$ . MW, 178. B.p. 240°.

*d-Amyl ester*: b.p. 266–8°/725 mm.  $D^{20}_D$  0.976.  $n_D$  1.4929.  $[\alpha]^{20}_D + 6.59^\circ$ .

*l-Menthyl ester*: b.p. 228–9°/36 mm., 197°/15 mm.  $D^{20}_D$  0.9931.  $[\alpha]^{20}_D - 87.59^\circ$ .

*p-Nitrobenzyl ester*: cryst. from EtOH.Aq. M.p. 86.6°.

*p-Bromphenacyl ester*: m.p. 108°.

*Anhydride*: cryst. from pet. ether. M.p. 71°. B.p. 230°/17 mm. Sol. most org. solvents.

*Chloride*: m.p. –23°. B.p. 219–20°/773 mm., 136–8°/31 mm., 109°/15 mm.  $D^{20}_D$  1.173.

*Bromide*: b.p. 136–7°/52 mm.

*Amide*: see *m*-Toluamide.

*Nitrile*: see *m*-Tolunitrile.

*Hydrazide*: plates from EtOH.Aq. M.p. 97°.

Adams, Ulich, *J. Am. Chem. Soc.*, 1920, **42**, 608.

Reuter, *Ber.*, 1884, **17**, 2028.

### *p*-Toluic Acid (4-Methylbenzoic acid).

Cryst. from hot  $H_2O$ . M.p. 181°. B.p. 274–5°. Very sol. EtOH, MeOH,  $Et_2O$ . Spar. sol.  $H_2O$ . Volatile in steam. Sublimes. Heat of comb.  $C_p$  927.4 Cal.,  $C_p$  926.8 Cal.  $k = 4.3 \times 10^{-5}$  at 25°. Dist. with CaO  $\rightarrow$  toluene. Ox.  $\rightarrow$  terephthalic acid. Na + amyl alcohol  $\rightarrow$  hexahydro-*p*-toluic acid.

*Me ester*: cryst. with intense unpleasant odour from MeOH.Aq. or pet. ether. M.p. 33.2°. B.p. 217°.

*Et ester*: b.p. 228°, 122°/22 mm., 110°/12 mm.  $D^{25}_D$  1.024.  $n^{18.2}_D$  1.5089.

*d-Amyl ester*: b.p. 271–2°.  $D^{20}_D$  0.982.  $n^{19.1}_D$  1.4975.  $[\alpha]^{20}_D + 6.67^\circ$ .

*Isoamyl ester*:  $C_{13}H_{18}O_2$ . MW, 206. B.p. 271°.

*l-Menthyl ester*: cryst. M.p. 40–1°. B.p. 196–8°/11 mm.  $[\alpha]_D^{20} - 89-93^\circ$ .

*Phenyl ester*: plates from EtOH. M.p. 83°.

*p-Nitrobenzyl ester*: cryst. from EtOH.Aq. M.p. 104–5°.

*p-Bromphenacyl ester*: m.p. 153°.

*Anhydride*: plates from MeOH, needles from EtOH. M.p. 95°. Stable to boiling  $H_2O$ .

*Chloride*: m.p. – 2 to – 1.5°. B.p. 214–16°, 102°/15 mm.

*Bromide*: b.p. 145–9°/42 mm.

*Amide*: see *p-Toluamide*.

*Nitrile*: see *p-Tolunitrile*.

*Imide*: needles from  $C_6H_6$ . M.p. 155°.

*Hydrazide*: plates from EtOH.Aq. M.p. 117°.

*Anilide*: needles or plates from EtOH. M.p. 147–8°.

*o-Nitroanilide*: yellow prisms from EtOH. M.p. 110°.

*p-Toluidide*: needles from EtOH. M.p. 165° (158–9°).

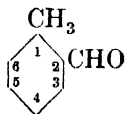
Herb, *Ann.*, 1890, 258, 10.

Gattermann, Schmidt, *Ann.*, 1888, 244, 51.

### $\alpha$ -Toluic Aldehyde.

See Phenylacetaldehyde.

### o-Toluic Aldehyde (2-Methylbenzaldehyde)



$C_8H_8O$

MW, 120

B.p. 197°, 94°/10 mm.  $D_4^{20}$  1.0386.  $n_D^{20}$  1.549.  $KCN + EtOH \rightarrow$  *o*-toluoin. Ox. in air  $\rightarrow$  *o*-toluic acid. Red.  $\rightarrow$  *o*-tolylcarbinol. Sol.  $H_2SO_4 \rightarrow$  greenish-orange sol.

*Oxime*: needles from pet. ether. M.p. 49°. Very sol. hot  $H_2O$ , EtOH,  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol. pet. ether. *N-Acetyl*: cryst. from  $Et_2O$ . M.p. 55–6°.

*Semicarbazone*: needles from amyl alcohol. M.p. 212° (196°).

*Hydrazone*: m.p. 97°.

*Phenylhydrazone*: m.p. 105–6°.

*p-Nitrophenylhydrazone*: red needles from EtOH. M.p. 222°.

2:4-Dinitrophenylhydrazone: orange-red plates from AcOH. M.p. 193–4°.

Rupe, Bernstein, *Helv. Chim. Acta*, 1930, 13, 460.

Stephen, *J. Chem. Soc.*, 1925, 1874.

Gattermann, *Ann.*, 1912, 393, 218.

Gattermann, Maffezoli, *Ber.*, 1903, 36, 4152.

### m-Toluic Aldehyde (3-Methylbenzaldehyde).

B.p. 199°, 93–4°/17 mm.  $D_4^{21.4}$  1.0189.  $n_D^{21.4}$  1.541. Oxidises in air to *m*-toluic acid.  $HNO_3 \rightarrow$  isophthalic acid.

*Semicarbazone*: needles from EtOH, plates from amyl alcohol. M.p. 223–4° (206°).

*Phenylhydrazone*: prisms from ligroin or EtOH.Aq. M.p. 90°.

Rupe, Bernstein, *Helv. Chim. Acta*, 1930, 13, 462.

Sommelet, *Compt. rend.*, 1913, 157, 853.

### p-Toluic Aldehyde (4-Methylbenzaldehyde).

Liq. with peppermint odour. B.p. 204–5°, 106°/10 mm.  $D_4^{26.7}$  1.0194.  $n_D^{26.6}$  1.547.  $KCN + EtOH \rightarrow$  4:4'-dimethylbenzoin. Oxidises in air to *p*-toluic acid. Forms cryst. bisulphite comp. Sol.  $H_2SO_4 \rightarrow$  orange-brown sol.

*Polymer*:  $(C_8H_8O)_x$ . Cryst. from  $C_6H_6$ . M.p. 215°. Insol. cold EtOH,  $Et_2O$ , AcOH.

*Di-Et acetal*:  $C_{12}H_{18}O_2$ . MW, 194. B.p. 116–19°/12 mm.  $D_4^{22}$  1.006.  $n_D^{22}$  1.47603.

$NH_3$  add. comp.: cryst. M.p. 43–4°.

*Oxime*: *syn.* M.p. 108–10°. Steam dist. of alk. sol.  $\rightarrow$  *anti.* *N-Acetyl*: m.p. 85°. *Anti*: m.p. 79–80°.

*Semicarbazone*: needles from EtOH, plates from amyl alcohol. M.p. 234° (215°).

*Hydrazone*: cryst. mass. M.p. 56°. B.p. 148°/12 mm.

*Phenylhydrazone*: plates from EtOH. M.p. 121° (108°).

*o-Nitrophenylhydrazone*: red needles from EtOH.Aq. M.p. 183°.

*m-Nitrophenylhydrazone*: m.p. 155°.

*p-Nitrophenylhydrazone*: dark red needles with green fluor. from EtOH. M.p. 200–5°.

2:4-Dinitrophenylhydrazone: orange-yellow cryst. from  $PhNO_2$ . M.p. 232.5–234.5°.

*p-Bromphenylhydrazone*: m.p. 162° decomp.

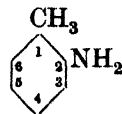
I.C.I., E.P., 397,124, (*Chem. Abstracts*, 1934, 28, 778).

Coleman, Craig, *Organic Syntheses*, 1932, XII, 80.

Hinkel, Ayling, Morgan, *J. Chem. Soc.*, 1932, 2793.

Gattermann, Koch, *Ber.*, 1897, 30, 1623.

### o-Toluidine (2-Aminotoluene, o-tolylamine)



$C_7H_9N$

MW, 107

Liq. Cryst. on cooling to two forms.  $\alpha$ -. M.p. about – 21°.  $\beta$ - (stable form). M.p. about

— 15.5°. B.p. 200.6°/754.6 mm., 121°/80 mm.  $D_{20}^{20}$  1.0053.  $n_D^{20}$  1.5688. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Specific heat 0.49 cal./gm. at 15–64°. Heat of comb.  $C_p$  963.8 Cal.,  $C_p$  969.93 Cal.  $k = 3.5 \times 10^{-10}$  at 25°. Volatile in steam.  $MnO_2 + H_2SO_4 \rightarrow$  toluquinone.  $KMnO_4 \rightarrow$  2:2'-dimethylazobenzene. Dil.  $H_2SO_4$  sol. +  $CrO_3$  in dil.  $H_2SO_4 \rightarrow$  blue col.  $\rightarrow$  reddish-violet col. on dilution. Forms Ca and Na derivs.

$C_7H_9N, C_6H_3(NO_2)_3 \cdot 1:3:5$ : light red needles. M.p. 125–7°.

$B, HCl$ : cryst. M.p. 215°. B.p. 242.2°.

$B, H_3PO_3$ : needles. M.p. 174°.

$B, HClO_3$ : plates from EtOH. Explodes at 88°.

$B_2, H_2PtBr_6$ : yellowish-red needles. M.p. 225–6° decomp.

$B, CH_3(COOH)_2$ : prisms. Decomp. at 108°.

$B, ClCH_2COOH$ : m.p. 95°.

$B_2, (COOH)_2$ : m.p. 167°.

$B, (COOH)_2$ : plates. M.p. 171°.

$B, (CH_2COOH)_2$ : prisms. Decomp. at 60°.

$B_2, ZnCl_2$ : cryst. from EtOH. M.p. 227°.

$B_2, ZnBr_2$ : cryst. from EtOH. M.p. 218°.

*N-Me*: see *N-Methyl-o-toluidine*.

*N-Di-Me*: see *Dimethyl-o-toluidine*.

*N-Et*: see *N-Ethyl-o-toluidine*.

*N-Di-Et*: see *Diethyl-o-toluidine*.

*N-Propyl*: see *N-Propyl-o-toluidine*.

*N-Butyl*:  $C_{11}H_{17}N$ . MW, 163. Oil with pleasant odour. B.p. 258–60°/771 mm.

*N-Isobutyl*: see *N-Isobutyl-o-toluidine*.

*N-Isoamyl*: see *N-Isoamyl-o-toluidine*.

*N-Allyl*:  $C_{10}H_{13}N$ . MW, 147. B.p. 225–30°.

*N-β-Hydroxyethyl*: see *N-β-Hydroxyethyl-o-toluidine*.

*N-Phenyl*: see *2-Methyldiphenylamine*.

*N-Diphenyl*: see *Diphenyl-o-toluidine*.

*N-Tolyl*: see *Ditolylamine*.

*N-Benzyl*: see *N-Benzyl-o-toluidine*.

*N-Dibenzyl*:  $C_{21}H_{21}N$ . MW, 287. Cryst. M.p. 42°. B.p. 223°/10 mm.  $D_4^{25}$  1.02347.  $n_D^{25}$  1.58324.

*N-Picryl*:  $C_{13}H_{10}O_6N_4$ . MW, 318. Orange-red prisms from EtOH–Me<sub>2</sub>CO. M.p. 164°.

*N-Formyl*: see *Formo-o-toluidide*.

*N-Acetyl*: see *Acet-o-toluidide*.

*N-Diacetyl*: *N-o-tolyldiacetamide*. Cryst. M.p. 18°. B.p. 200.5–201°/100 mm., 144–5°/11 mm.

*N-Benzoyl*: *benz-o-toluidide*. Needles from AcOEt–Me<sub>2</sub>CO. M.p. 145–6°.

*N-Dibenzoyl*: *N-o-tolyldibenzamide*. Prisms from EtOH. M.p. 111–12°.

*N-p-Toluenesulphonyl*: m.p. 185.5–186.2°.

*N-1-Naphthalenesulphonyl*: m.p. 237°.

*Picrate*: cryst. from EtOH. M.p. 212–15° decomp.

Tanner, Lasselle, *J. Am. Chem. Soc.*, 1926, **48**, 2163.

Courtot, Petitcolas, *Bull. soc. chim.*, 1926, **39**, 452.

Blanksma, *Rec. trav. chim.*, 1909, **28**, 109.

**m-Toluidine** (3-Aminotoluene, m-tolylamine).

Liq. Cryst. on strong cooling. M.p. — 43.6°. B.p. 203.2°.  $D_{20}^{20}$  0.990.  $n_D^{20}$  1.56859. Sol. EtOH, Et<sub>2</sub>O. Very spar. sol. H<sub>2</sub>O. Heat of comb.  $C_p$  964.6 Cal.,  $C_p$  965.6 Cal.  $k = 5.5 \times 10^{-10}$  at 25°. Volatile in steam. Ox.  $\rightarrow$  toluquinone. Sol. in dil.  $H_2SO_4$  +  $CrO_3$  in dil.  $H_2SO_4 \rightarrow$  yellowish-brown col. Sol. in dil.  $H_2SO_4$  +  $HNO_3 \rightarrow$  dark red col.

$C_7H_9N, C_6H_3(NO_2)_3 \cdot 1:3:5$ : light red needles. M.p. 93°.

$B, HCl$ : plates from H<sub>2</sub>O. M.p. 228°. B.p. 249.8°.

$B, HClO_4$ : m.p. 200° decomp.

$B_2, H_2PtBr_6$ : red scales. M.p. 266° decomp.

$B, CH_3(COOH)_2$ : prisms. Decomp. at 93°.

$B, (CH_2COOH)_2$ : columns. Decomp. at 121°.

*N-Me*: see *N-Methyl-m-toluidine*.

*N-Di-Me*: see *Dimethyl-m-toluidine*.

*N-Et*: see *N-Ethyl-m-toluidine*.

*N-Di-Et*: see *Diethyl-m-toluidine*.

*N-Phenyl*: see *3-Methyldiphenylamine*.

*N-Diphenyl*: see *Diphenyl-m-toluidine*.

*N-Tolyl*: see *Ditolylamine*.

*N-Benzyl*: see *N-Benzyl-m-toluidine*.

*N-Dibenzyl*:  $C_{21}H_{21}N$ . MW, 287. Cryst. M.p. 78°. B.p. 229°/10 mm.

*N-Picryl*: two forms. α-. Yellow prisms from EtOH–HCl. M.p. 130°. β-. Orange-red needles from alc. NH<sub>3</sub>. M.p. 129.5°.

*N-Formyl*: see *Formo-m-toluidide*.

*N-Acetyl*: see *Acet-m-toluidide*.

*N-Benzoyl*: *benz-m-toluidide*. Prisms from EtOH.Aq. M.p. 125°.

*N-Dibenzoyl*: *N-m-tolyldibenzamide*. Needles from EtOH. M.p. 140–1°.

*N-p-Toluenesulphonyl*: m.p. 171–172.5°.

*N-1-Naphthalenesulphonyl*: m.p. 195–6°.

Buchka, Schachtebeck, *Ber.*, 1889, **22**, 840.

Blanksma, *Rec. trav. chim.*, 1906, **25**, 370. Ehrlich, *Ber.*, 1882, **15**, 2011.

**p-Toluidine** (4-Aminotoluene, p-tolylamine).

Cryst. + 1H<sub>2</sub>O from H<sub>2</sub>O or EtOH.Aq. M.p. 42°, anhyd. 44.5–45°. Anhyd. in moist air  $\rightarrow$  monohydrate. B.p. 200.3°, 133.7°/100 mm.,

100-2°/25 mm., 82-2°/10 mm. Sol. 285 parts H<sub>2</sub>O at 11-5°. Very sol. EtOH.Aq., MeOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CS<sub>2</sub>. Volatile in steam. D<sub>4</sub><sup>20</sup> 0.973. n<sub>D</sub><sup>20</sup> 1.55324. Heat of comb. C<sub>r</sub> 957.9 Cal., C<sub>p</sub> 958.8 Cal., C<sub>p</sub> (liq.) 973.5 Cal.  $k = 1.48 \times 10^{-9}$  at 25°. Alk. KMnO<sub>4</sub> → 4 : 4'-dimethylazobenzene. Acid KMnO<sub>4</sub> → *p*-nitrosotoluene. Reduces alc. NH<sub>3</sub>.AgNO<sub>3</sub> in the cold. Dil. H<sub>2</sub>SO<sub>4</sub> sol. + HNO<sub>3</sub> → blue → violet → red → brown col. Slightly acid sol. + FeCl<sub>3</sub> → pale yellow → red col. Forms metallic derivs. with Ca and Na.

*B.HCl*: needles from AcOH-Et<sub>2</sub>O. M.p. 243°. B.p. 257-5°.

*B<sub>2</sub>H<sub>2</sub>PtBr<sub>6</sub>*: yellowish-red cryst. M.p. 268-9°.

*B.ClCH<sub>2</sub>COOH*: needles. M.p. 101-2°.

*B.Cl<sub>2</sub>CHCOOH*: needles. M.p. 140-1°.

*B.Cl<sub>3</sub>C·COOH*: cryst. M.p. 137°.

*B<sub>2</sub>(COOH)<sub>2</sub>*: m.p. 183-4°.

*B<sub>1</sub>(COOH)<sub>2</sub>*: m.p. 178°.

*B.CH<sub>2</sub>(COOH)<sub>2</sub>*: m.p. 114°.

*B.(CH<sub>2</sub>COOH)<sub>2</sub>*: m.p. 123-4°.

*B.C<sub>6</sub>H<sub>5</sub>COOH*: m.p. 52-5°.

*B<sub>2</sub>.AgNO<sub>3</sub>*: plates. M.p. 101° decomp.

*N-Me*: see *N*-Methyl-*p*-toluidine.

*N-Di-Me*: see Dimethyl-*p*-toluidine.

*N-Et*: see *N*-Ethyl-*p*-toluidine.

*N-Di-Et*: see Diethyl-*p*-toluidine.

*N-Propyl*: see *N*-Propyl-*p*-toluidine.

*N-Isopropyl*: see *N*-Isopropyl-*p*-toluidine.

*N-Butyl*: C<sub>11</sub>H<sub>17</sub>N. MW, 163. Oil. B.p. 264-5°/766 mm. Volatile in steam. *B.HCl*: needles or prisms from EtOH. M.p. 150-1°.

*N-Dibutyl*: C<sub>15</sub>H<sub>25</sub>N. MW, 219. Oil. B.p. 282-4°/764 mm. *Picrate*: yellow cryst. from Et<sub>2</sub>O-pet. ether. M.p. 131-2°.

*N-Isobutyl*: see *N*-Isobutyl-*p*-toluidine.

*N-Allyl*: C<sub>10</sub>H<sub>13</sub>N. MW, 147. Oil. B.p. 232-4°. *B.HCl*: m.p. 131-2°.

*N-β-Hydroxyethyl*: see *N*-β-Hydroxyethyl-*p*-toluidine.

*N-Phenyl*: see 4-Methyldiphenylamine.

*N-Tolyl*: see Ditolylamine.

*N-Benzyl*: see *N*-Benzyl-*p*-toluidine.

*N-Dibenzyl*: C<sub>21</sub>H<sub>21</sub>N. MW, 287. Cryst. M.p. 56°. B.p. 233°/11 mm. D<sub>4</sub><sup>25</sup> 1.03721. n<sub>D</sub><sup>25</sup> 1.60109.

*N-Picryl*: two forms. α-. Yellow needles from Me<sub>2</sub>CO, CHCl<sub>3</sub>, CCl<sub>4</sub> or C<sub>6</sub>H<sub>6</sub>. M.p. 164°. β-. Dark red needles from Py. M.p. 164°.

*N-Bornyl*: C<sub>17</sub>H<sub>25</sub>N. MW, 243. Needles. M.p. 33°. B.p. 162°/3 mm. Very sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. *B.HCl*: cryst. powder. M.p. 214° decomp.

*N-Formyl*: see Formo-*p*-toluidide.

*N-Acetyl*: see Acet-*p*-toluidide.

*N-Diacetyl*: *N*-*p*-tolylldiacetamide. Cryst. M.p. 48°. B.p. 160-1°/15 mm.

*N-Benzoyl*: benz-*p*-toluidide. Needles from EtOH. M.p. 158°. B.p. 232°. *N-Nitroso*: needles from Me<sub>2</sub>CO.Aq. M.p. 75° decomp.

*N-Dibenzoyl*: *N*-*p*-tolylldibenzamide. Prisms from EtOH. M.p. 142-4°.

*N-p-Toluenesulphonyl*: m.p. 193-194.5°.

*N-1-Naphthalenesulphonyl*: m.p. 181°.

*Picrate*: m.p. 180-1° decomp.

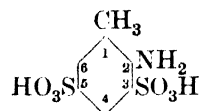
Blanksma, *Rec. trav. chim.*, 1909, **28**, 109; 1906, **25**, 370.

Popov, *Chem. Abstracts*, 1934, **28**, 1671.

Kock, *Ber.*, 1887, **20**, 1568.

Graebe, *Ber.*, 1901, **34**, 1778.

**o-Toluidine-3 : 5-disulphonic Acid**



C<sub>7</sub>H<sub>9</sub>O<sub>6</sub>NS<sub>2</sub>

MW, 267

Needles + 1½H<sub>2</sub>O from H<sub>2</sub>O. Decomp. at 240°. Very sol. H<sub>2</sub>O, EtOH. Insol. C<sub>6</sub>H<sub>6</sub>.

*Dichloride*: C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>NCl<sub>2</sub>S<sub>2</sub>. MW, 304. Cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 153°.

*Diamide*: cryst. from EtOH.Aq. M.p. 188°.

Pollak, Pollak, Riesz, *Monatsh.*, 1931, **58**, 128.

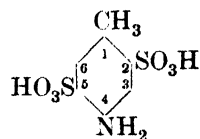
Neville, Winther, *Ber.*, 1882, **15**, 2992.

**o-Toluidine-4 : 5-disulphonic Acid.**

Needles. Spar. sol. Forms spar. sol. Ba salt.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 745.

**p-Toluidine-2 : 5-disulphonic Acid**



C<sub>7</sub>H<sub>9</sub>O<sub>6</sub>NS<sub>2</sub>

MW, 267

Needles + 2½H<sub>2</sub>O. Decomp. at 290°. Very sol. H<sub>2</sub>O. Spar. sol. EtOH. Forms spar. sol. Ba salt.

*Dichloride*: C<sub>7</sub>H<sub>7</sub>O<sub>4</sub>NCl<sub>2</sub>S<sub>2</sub>. MW, 304. Yellow prisms from CHCl<sub>3</sub>. M.p. 156°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. *N-Acetyl*: cryst. from pet. ether. M.p. 125°. *N-Chlor-acetyl*: cryst. from pet. ether. M.p. 118-19°.

*Diamide*: C<sub>7</sub>H<sub>11</sub>O<sub>4</sub>N<sub>2</sub>S<sub>2</sub>. MW, 265. Needles from H<sub>2</sub>O. M.p. 257°. Sol. H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO.

*Dianilide*: plates from EtOH.Aq. M.p. 196–7°.

Pollak, Pollak, Riesz, *Monatsh.*, 1931, **58**, 126.

Riesz, Pollak, Wittels, *Ann.*, 1931, **487**, 267.

Lustig, Katscher, *Monatsh.*, 1927, **48**, 93.

**p-Toluidine-2 : 6-disulphonic Acid.**

Prisms + H<sub>2</sub>O. Very sol. H<sub>2</sub>O. Spar. sol. EtOH. Ppd. by EtOH from H<sub>2</sub>O.

Kornatzki, *Ann.*, 1883, **221**, 198.

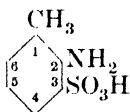
**p-Toluidine-3 : 5-disulphonic Acid.**

Needles + 2H<sub>2</sub>O, aggregates of cryst. + 1H<sub>2</sub>O. Anhyd. at 120°. Decomp. at 200°. Very sol. H<sub>2</sub>O. Mod. sol. EtOH. Decomp. with H<sub>2</sub>O at 140° → *p*-toluidine-3-sulphonic acid.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 734.

Richter, *Ann.*, 1885, **230**, 315.

**o-Toluidine-3-sulphonic Acid**



C<sub>7</sub>H<sub>9</sub>O<sub>3</sub>NS

MW, 187

Needles. Very sol. hot H<sub>2</sub>O. Warm with aq. FeCl<sub>3</sub> → reddish-yellow col.

Pechmann, *Ann.*, 1874, **173**, 215.

**o-Toluidine-4-sulphonic Acid.**

Needles or prisms + 1H<sub>2</sub>O. Anhyd. over H<sub>2</sub>SO<sub>4</sub>. Sol. 104 parts H<sub>2</sub>O at 11°. Insol. EtOH.  $k = 2.5 \times 10^{-4}$  at 25°. FeCl<sub>3</sub> → dark violet col. NaOH fusion → anthranilic acid.

*Fluoride*: C<sub>7</sub>H<sub>8</sub>O<sub>2</sub>NFS. MW, 189. Needles from EtOH.Aq. M.p. 96–7°. Very sol. Me<sub>2</sub>CO. Sol. Et<sub>2</sub>O, AcOEt, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, CS<sub>2</sub>, pet. ether. Spar. sol. H<sub>2</sub>O. *N-Acetyl*: needles from EtOH. M.p. 188.5–189.5°.

*Chloride*: C<sub>7</sub>H<sub>8</sub>O<sub>2</sub>NCIS. MW, 205.5. *N-Acetyl*: prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 144°. *N-Benzoyl*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 196°.

*Amide*: C<sub>7</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>S. MW, 186. Columns. M.p. 176°. Very sol. hot H<sub>2</sub>O. Spar. sol. EtOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. *B.HCl*: needles. M.p. 240°.

*N-Me*: C<sub>8</sub>H<sub>11</sub>O<sub>3</sub>NS. MW, 201. Plates from hot H<sub>2</sub>O. Sol. 60 parts H<sub>2</sub>O at 15°.

*N-Di-Et*: C<sub>11</sub>H<sub>17</sub>O<sub>3</sub>NS. MW, 243. Plates + 1H<sub>2</sub>O from H<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O, EtOH.

*N-Benzoyl*: plates. M.p. 203°.

I.G., D.R.P., 573,193, (*Chem. Zentr.*, 1933, II, 445).

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 26.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 745.

**o-Toluidine-5-sulphonic Acid.**

Plates or columns from 1H<sub>2</sub>O + H<sub>2</sub>O. Anhyd. at 120° over H<sub>2</sub>SO<sub>4</sub>. Sol. 32 parts H<sub>2</sub>O at 19°.  $k = 7.53 \times 10^{-4}$  at 25°. Characteristic col. with PbO<sub>2</sub> in aq. sol.

*N-Me*: needles from H<sub>2</sub>O. Sol. H<sub>2</sub>O.

*N-Di-Me*: C<sub>9</sub>H<sub>13</sub>O<sub>3</sub>NS. MW, 215. Prisms from H<sub>2</sub>O. Very sol. hot H<sub>2</sub>O. Insol. EtOH.

Schultz, Lucas, *J. Am. Chem. Soc.*, 1927, **49**, 299.

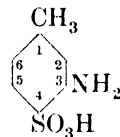
Neville, Winther, *Ber.*, 1880, **13**, 1941.

**o-Toluidine-6-sulphonic Acid.**

Needles. Sol. 293 parts H<sub>2</sub>O at 22°.

Pagel, *Ann.*, 1875, **176**, 305.

**m-Toluidine-4-sulphonic Acid**



C<sub>7</sub>H<sub>9</sub>O<sub>3</sub>NS

MW, 187

Needles + H<sub>2</sub>O. Sol. 715 parts H<sub>2</sub>O at 16°.

Hayduck, *Ann.*, 1874, **174**, 350.

**m-Toluidine-6-sulphonic Acid.**

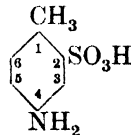
Plates. Chars above 275°. Spar. sol. H<sub>2</sub>O.  $k = 3.57 \times 10^{-4}$  at 25°.

*NH<sub>4</sub> salt*: m.p. 190°.

Quilico, *Gazz. chim. ital.*, 1926, **56**, 626.

Seyewetz, Bloch, *Bull. soc. chim.*, 1907, **1**, 327.

**p-Toluidine-2-sulphonic Acid**



C<sub>7</sub>H<sub>9</sub>O<sub>3</sub>NS

MW, 187

Prisms + 1H<sub>2</sub>O. Decomp. at high temps. Sol. 220 parts H<sub>2</sub>O at 20°. Insol. EtOH.  $k = 4.08 \times 10^{-5}$  at 25°. FeCl<sub>3</sub> → red col. Reduces NH<sub>3</sub>.AgNO<sub>3</sub> on warming.

*Fluoride*: C<sub>7</sub>H<sub>8</sub>O<sub>2</sub>NFS. MW, 189. Yellow cryst. from EtOH. M.p. 62°. Sol. Et<sub>2</sub>O, AcOEt, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O. *N-Acetyl*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 120–1°.

**Chloride**:  $C_7H_8O_2NCIS$ . MW, 205.5. N-Acetyl: prisms from  $Me_2CO$ . Aq. M.p. 124°. N-Chloracetyl: plates from petrol. M.p. 87°.

**Amide**:  $C_7H_{10}O_2N_2S$ . MW, 186. Needles or plates. M.p. 164°. Sol. hot  $H_2O$ , EtOH. N-Acetyl: cryst. from  $H_2O$ . M.p. 242°. N-Chloracetyl: needles from  $H_2O$ . M.p. 231°.

N-Me:  $C_8H_{11}O_3NS$ . MW, 201. Plates. Sol.  $H_2O$ . Insol. org. solvents. Decomp. on boiling aq. sol.

N-Et:  $C_9H_{13}O_3NS$ . MW, 215. Yellow prisms +  $1H_2O$  from  $H_2O$ . Spar. sol.  $H_2O$ . Very spar. sol. EtOH.

N-Di-Et:  $C_{11}H_{17}O_3NS$ . MW, 243. Prisms +  $1H_2O$  from  $H_2O$ . Does not melt. Spar. sol.  $H_2O$ . Insol. Et<sub>2</sub>O.

N-Isopropyl:  $C_{10}H_{15}O_3NS$ . MW, 229. Prisms from  $H_2O$ . Does not melt below 300°. Spar. sol. EtOH.

N-Acetyl: needles +  $2H_2O$  from dil. HCl. Anilide: leaflets from EtOH. M.p. 230°.

Anilide: m.p. 146-7°.

Pollak, Pollak, Riesz, *Monatsh.*, 1931, **58**, 125.

Steinkopf, *J. prakt. Chem.*, 1927, **117**, 37.

Johnson, Smiles, *J. Chem. Soc.*, 1923, **123**, 2385.

**p-Toluidine-3-sulphonic Acid.**

Needles +  $\frac{1}{2}H_2O$ . Sol. 214 parts  $H_2O$ .  $k = 8.5 \times 10^{-4}$  at 25°. Aq. sol. +  $PbO_2 \rightarrow$  red col. Decomp. with  $H_2O$  at 130°. Gives Ag salt, spar. sol.  $H_2O$  at 100°.

N-Me:  $C_8H_{11}O_3NS$ . MW, 201. Prisms from  $H_2O$ . Decomp. on heating.

N-Di-Et: prisms +  $1H_2O$  from  $H_2O$ . M.p. 243°. Sol.  $H_2O$ . Insol. Et<sub>2</sub>O,  $C_6H_6$ . K salt: plates +  $1\frac{1}{2}H_2O$ . M.p. 297°.

N-Di-isopropyl:  $C_{13}H_{21}O_3NS$ . MW, 271. Cryst. +  $2H_2O$  from  $H_2O$ . M.p. 222-3°.

Leitch, E.P., 257,979, (*Chem. Zentr.*, 1927, I, 1745).

Witt, Uerményi, *Ber.*, 1913, **46**, 301.

Wynne, Bruce, *J. Chem. Soc.*, 1898, **73**, 738.

**Toluidinoacetic Acid.**

See Tolyglycine.

**Toluidinobenzoic Acid.**

See Methylphenylamine-carboxylic Acid.

**2-Toluidinoethyl Alcohol.**

See N-β-Hydroxyethyltoluidine.

**Toluidinophenol.**

See Hydroxy-methylphenylamine.

**Tolunitranilic Acid.**

See 5-Nitro-3 : 6-dihydroxytoluquinone.

**o-Tolunitrile (o-Cyanotoluene)**



$C_8H_7N$

MW, 117

Liq. M.p. - 13 to - 14°. B.p. 205.2°, 90°/15 mm., 82.3°/11 mm.  $D_{25}^{20}$  0.9912.  $n_D^{20}$  1.52720. Heat of comb.  $C_r$  1030 Cal.,  $C_p$  1030.7 Cal. Red.  $\rightarrow$  o-xylylamine. Hyd.  $\rightarrow$  o-toluic acid.

Clarke, Read, *Organic Syntheses*, Collective Vol. I, 500.

**m-Tolunitrile (m-Cyanotoluene).**

Liq. M.p. - 23.5 to - 23°. B.p. 210-12°, 84.5°/10 mm.  $D_{20}^{20}$  1.0316. Red.  $\rightarrow$  m-xylylamine.  $H_2O_2 \rightarrow$  m-toluamide.

Bayer, D.R.Ps., 259,363, 259,364, (*Chem. Zentr.*, 1913, I, 1741).

Buchka, Schachtebeck, *Ber.*, 1889, **22**, 841.

**p-Tolunitrile (p-Cyanotoluene).**

Needles from EtOH. M.p. 29°. B.p. 217.6°, 90.5-91°/11 mm.  $D_{30}^{30}$  0.9805.  $KMnO_4 \rightarrow$  terephthalamic acid.  $H_2O_2 \rightarrow$  p-toluamide.

Clarke, Read, *Organic Syntheses*, Collective Vol. I, 500.

**Toluoin.**

See Dimethylbenzoin.

**Toluylenediamine.**

See Tolylenediamine.

**Toluquinaldine.**

See 2 : 6-, 2 : 7-, and 2 : 8-Dimethylquinoline.

**Toluquinhydrone.**

See under Toluquinone.

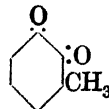
**Toluquinol.**

See Toluhydroquinone.

**Toluquinoline.**

See 6-, 7-, and 8-Methylquinoline.

**o-Toluquinone (3-Methyl-o-benzoquinone)**



$C_7H_6O_2$

MW, 122

Dark red prisms and needles from Et<sub>2</sub>O or pet. ether. Sol. Et<sub>2</sub>O  $\rightarrow$  green sol. Warm, or standing in Et<sub>2</sub>O  $\rightarrow$  dimer.

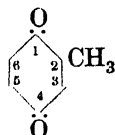
Dimer:  $C_{14}H_{12}O_4$ . MW, 244. Yellow prisms and plates from  $CHCl_3$ -pet. ether. M.p. 194-5°.

Very sol.  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ , pet. ether. Gives no quinone reactions.

*Dioxime*: yellowish-brown needles from  $\text{H}_2\text{O}$ . M.p. about  $140^\circ$  decomp. Sol.  $\text{EtOH}$ ,  $\text{AcOH}$ . Mod. sol.  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

Willstätter, Müller, *Ber.*, 1911, **44**, 2178.

**Toluquinone** (p-Toluquinone, methylquinone, 2-methyl-p-benzoquinone)



$\text{C}_7\text{H}_6\text{O}_2$

MW, 122

Yellow plates or needles. M.p.  $69^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Volatile in steam. Sublimes. Heat of comb.  $\text{C}_c$  805.0 Cal.,  $\text{C}_p$  805.3 Cal. Mod. conc.  $\text{H}_2\text{SO}_4 \rightarrow$  polymer. Red.  $\rightarrow$  toluhydroquinone. Triphenylmagnesium chloride  $\rightarrow$  toluquinhydrone. Aq. sol. + alkali  $\rightarrow$  brownish-red col.

*Polymer*: powder. Does not melt below  $300^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Sol.  $\text{AcOH}$ . Spar. sol.  $\text{H}_2\text{O}$ . Insol.  $\text{C}_6\text{H}_6$ .

*Toluhydroquinone comp.*: toluquinhydrone.  $\text{C}_{14}\text{H}_{14}\text{O}_4$ . MW, 246. Black needles. M.p.  $96-7^\circ$ .

1-Oxime: see 6-Nitroso-m-cresol.

4-Oxime: see 5-Nitroso-o-cresol.

1-Oxime-4-imide: see 6-Nitroso-m-toluidine.

4-Oxime-1-imide: see 5-Nitroso-o-toluidine.

*Dioxime*: needles. Decomp. at  $234^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol. ligroin. N-Diacetyl: needles from  $\text{EtOH}$ . Aq. M.p.  $120^\circ$ . N-Benzoyl: yellow cryst. from  $\text{EtOH}$ . M.p.  $180^\circ$  decomp.

1-Chloroimide:  $\text{C}_7\text{H}_6\text{ONCl}$ . MW, 155.5. Yellow columns from  $\text{EtOH}$ . M.p.  $75^\circ$ .

4-Chloroimide: yellow needles. M.p.  $87-8^\circ$ .

Dichloroimide:  $\text{C}_7\text{H}_6\text{N}_2\text{Cl}_2$ . MW, 189. Yellow needles. M.p.  $74^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ . Sol.  $\text{EtOH}$ .

1-Imide-4-semicarbazone: monohydrated reddish-brown needles from  $\text{Me}_2\text{CO}-\text{C}_6\text{H}_6$ . M.p.  $85-6^\circ$  decomp.

4-Semicarbazone: yellow needles from  $\text{EtOH}$ . M.p.  $178-9^\circ$ .

1:4-Disemicarbazone: orange-red. M.p.  $240^\circ$  decomp.

4-Phenylsemicarbazone: dark red cryst. from  $\text{EtOH}$ . M.p.  $198-9^\circ$  decomp.

1:4-Diphenylsemicarbazone: red. cryst. powder. Decomp. at  $246^\circ$ .

Di-2:4-dinitrophenylhydrazones: greyish-black needles from  $\text{PhNO}_2$ . M.p.  $269^\circ$ .

4-Benzoylphenylhydrazones: yellow plates from  $\text{C}_6\text{H}_6$ . M.p.  $151^\circ$ .

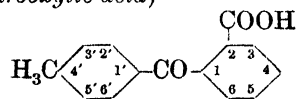
Clark, *Am. Chem. J.*, 1892, **14**, 565.

Borsche, Müller, Bodenstein, *Ann.*, 1929, **472**, 214.

**Toluquinoxaline.**

See 6-Methylquinoxaline.

**2-p-Toluybenzoic Acid** (4'-Methylbenzophenone-2-carboxylic acid)



$\text{C}_{15}\text{H}_{12}\text{O}_3$

MW, 240

Prisms +  $1\text{H}_2\text{O}$  from  $\text{EtOH}$ -toluene. M.p.  $146^\circ$  ( $139-40^\circ$ ). Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ , boiling toluene. Spar. sol.  $\text{H}_2\text{O}$ . Ox.  $\rightarrow$  benzophenone-2:4'-dicarboxylic acid.

*Me ester*:  $\text{C}_{16}\text{H}_{14}\text{O}_3$ . MW, 254. Plates from  $\text{MeOH}$ . M.p.  $61^\circ$  ( $53^\circ$ ). Sol.  $\text{EtOH}$ ,  $\text{C}_6\text{H}_6$ . Pale yellow sol. in conc.  $\text{H}_2\text{SO}_4$ .

*Et ester*:  $\text{C}_{17}\text{H}_{16}\text{O}_3$ . MW, 268. Plates. M.p.  $68-9^\circ$ .

*Amide*:  $\text{C}_{15}\text{H}_{13}\text{O}_2\text{N}$ . MW, 239. Needles from  $\text{H}_2\text{O}$ . M.p.  $175-6^\circ$ . Sol.  $\text{MeOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{AcOH}$ . Spar. sol.  $\text{C}_6\text{H}_6$ , ligroin.

Friedel, Crafts, *Bull. soc. chim.*, 1881, **35** 505.

Heller, Schülke, *Ber.*, 1908, **41**, 3632.

Fieser, *Organic Syntheses*, Collective Vol. I, 503.

Limpricht, *Ann.*, 1898, **299**, 306.

**4-p-Toluybenzoic Acid** (4'-Methylbenzophenone-4-carboxylic acid).

Needles from  $\text{MeOH}$ . M.p.  $228^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

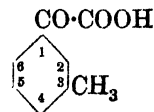
*Me ester*: needles from  $\text{MeOH}$ . M.p.  $126^\circ$ .

*Chloride*:  $\text{C}_{15}\text{H}_{11}\text{O}_2\text{Cl}$ . MW, 258.5. Cryst. from pet. ether. M.p.  $110^\circ$ .

*Amide*: prisms from  $\text{EtOH}$ . M.p.  $196^\circ$ .

Limpricht, Claus, *Ann.*, 1900, **312**, 92.

**m-Toluyformic Acid** (m-Tolylglyoxylic acid,  $\alpha$ -keto-m-tolylacetic acid, 3-methylbenzoylformic acid)



$\text{C}_9\text{H}_8\text{O}_3$

MW, 164

Colourless needles from  $\text{C}_6\text{H}_6$ -pet. ether. M.p.  $78-82^\circ$ . B.p.  $148-50^\circ/15-16\text{ mm}$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . Decomp. on dist. under atm. press.  $\rightarrow$  m-toluic acid + m-toluic



aldehyde.  $\text{H}_2\text{SO}_4$  + thiophene on  $\text{C}_6\text{H}_6$  sol.  $\rightarrow$  red  $\rightarrow$  bluish-violet col.

*Me ester*:  $\text{C}_{10}\text{H}_{10}\text{O}_3$ . MW, 178. B.p. 246–50°/763 mm. slight decomp., 137–8°/11–12 mm.

*Et ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_3$ . MW, 192. Oil with pleasant odour. B.p. 250–5° part. decomp., 141–2°/11–12 mm.

*Phenylhydrazone*: yellow needles from  $\text{AcOH.Aq}$ . M.p. 158°.

Posner, Heumann, *Ber.*, 1923, 56, 1624.

**p-Toluylic Acid** (*p-Tolylglyoxylic acid*,  $\alpha$ -*keto-p-tolylacetic acid*, 4-methylbenzoylformic acid).

Needles from ligroin. M.p. 97°. B.p. 164°/10 mm. Sol. most org. solvents. Spar. sol. hot  $\text{H}_2\text{O}$ . Dist. at ord. press.  $\rightarrow$  *p*-toluic acid + *p*-toluic aldehyde. Hot  $\text{H}_2\text{SO}_4 \rightarrow$  *p*-toluic acid. Ox.  $\rightarrow$  *p*-toluic acid + terephthalic acid. Red.  $\rightarrow$  *p*-tolylglycollic acid + *p*-tolylacetic acid.

*Et ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_3$ . MW, 192. Liq. B.p. 260–70°, 154–6°/18 mm. *Phenylhydrazone*: cryst. from ligroin–pet. ether. M.p. 94°.

*Amide*:  $\text{C}_9\text{H}_9\text{O}_2\text{N}$ . MW, 163. Prisms from  $\text{EtOH}$ . M.p. 160°.

*Nitrile*:  $\text{C}_8\text{H}_7\text{ON}$ . MW, 145. Prisms. M.p. 92°. Insol.  $\text{H}_2\text{O}$ . *Oxime*: leaflets from  $\text{H}_2\text{O}$ . M.p. 117°.

*Phenylhydrazone*: yellow needles from  $\text{C}_6\text{H}_6$ –ligroin. M.p. 145–6°.

Avogadro, *Gazz. chim. ital.*, 1923, 53, 698.

Auwers, *Ber.*, 1911, 44, 600.

Bouveault, *Bull. soc. chim.*, 1897, 17, 363, 367.

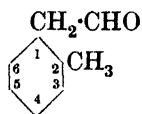
### Toluylic Acid.

See Phenylacetic Acid.

### Tolylphenol.

See 2', and 4'-Hydroxy-4-methylbenzophenone.

### o-Tolylacetaldehyde



$\text{C}_9\text{H}_{10}\text{O}$

MW, 134

Liq. with odour of jasmine. B.p. 219–21°/742 mm., 142–3°/90 mm., 92°/10 mm.  $\text{D}_4^{20}$  1.0241. Misc. with  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. misc. with  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin.

*Oxime*: needles. M.p. 102–3°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin.

Spath, *Monatsh.*, 1915, 36, 8.

### m-Tolylacetaldehyde.

Pleasant-smelling, pale yellow oil. B.p. 99–100°/18 mm. Reduces  $\text{NH}_3\text{.AgNO}_3$ . Hot alkalis  $\rightarrow$  resin.

*Benzoylhydrazone*: needles from  $\text{EtOH.Aq}$ . M.p. 129–30°.

*m-Nitrobenzoylhydrazone*: yellow needles from  $\text{EtOH.Aq}$ . M.p. 115–16°.

Curtius, Marangolo, *J. prakt. Chem.*, 1916, 94, 337.

### p-Tolylacetaldehyde.

Needles. M.p. about 40°. B.p. 221–2°, 96°/10 mm., 80–2°/3 mm. Very sol.  $\text{Et}_2\text{O}$ . Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ . Slowly oxidises in air to *p*-tolylacetic acid. Readily decomp. by acids. Electrolytic red.  $\rightarrow$   $\beta$ -*p*-tolylethyl alcohol. Forms bisulphite comp.

*Oxime*: prisms from  $\text{Et}_2\text{O}$ . M.p. 126–126.5°. Very sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Sol.  $\text{C}_6\text{H}_6$ , ligroin. Insol.  $\text{H}_2\text{O}$ .

*Semicarbazone*: cryst. from  $\text{EtOH}$ . M.p. about 208°.

Knorr, Weissenborn, Laage, U.S.P., 1,899,340, (*Chem. Zentr.*, 1932, II, 2747); D.R.P., 591,452, (*Chem. Zentr.*, 1932, II, 2748).

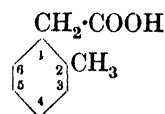
Spath, *Ber.*, 1914, 47, 767.

Auwers, *Ber.*, 1906, 39, 3761.

### Tolyl acetate.

See under Cresol.

**o-Tolylacetic Acid** (2-Methylphenylacetic acid)



$\text{C}_9\text{H}_{10}\text{O}_2$

MW, 150

Needles from  $\text{H}_2\text{O}$ . M.p. 88–9°. Very sol. hot  $\text{H}_2\text{O}$ . Ox.  $\rightarrow$  phthalic acid. Electrolytic red.  $\rightarrow$   $\beta$ -*o*-tolylethyl alcohol.

*Amide*:  $\text{C}_9\text{H}_{11}\text{ON}$ . MW, 149. Plates from  $\text{H}_2\text{O}$ . M.p. 161°. Very sol. hot  $\text{EtOH}$ . Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ . Sublimes.

*Nitrile*: *o*-tolubenzyl cyanide, *o*-xylyl cyanide.  $\text{C}_8\text{H}_9\text{N}$ . MW, 131. Liq. B.p. 244°.  $\text{D}_4^{20}$  1.0156.

Schorigin, *Ber.*, 1910, 43, 1941.

Radziszewski, Wispek, *Ber.*, 1885, 18, 1281.

**m-Tolylacetic Acid** (3-Methylphenylacetic acid).

Needles. M.p. 61°. Very sol. hot  $\text{H}_2\text{O}$ . Electrolytic red.  $\rightarrow$   $\beta$ -*m*-tolylethyl alcohol.

*Me ester*:  $C_{10}H_{12}O_2$ . MW, 164. B.p. 228–9°.  $D_{17.5}^{20}$  1.044.

*Et ester*:  $C_{11}H_{14}O_2$ . MW, 178. B.p. 237–8°.  $D_{17.5}^{20}$  1.018.

*Amide*: needles from  $H_2O$ . M.p. 141°. Sol. hot EtOH. Spar. sol. cold  $H_2O$ ,  $Et_2O$ . Sublimes in plates.

*Nitrile*: *m*-tolubenzyl cyanide, *m*-xylyl cyanide. B.p. 240–1°.  $D_{25}^{20}$  1.0022.

Radziszewski, Wispek, *Ber.*, 1885, **18**, 1282.

Seńkowski, *Monatsh.*, 1888, **9**, 855.

**p-Tolylacetic Acid** (4-Methylphenylacetic acid).

Needles or plates from  $H_2O$ . M.p. 94°. B.p. 265–7°. Sol. hot  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Sublimes. Electrolytic red.  $\rightarrow$   $\beta$ -*p*-tolylethyl alcohol.

*Et ester*: liq. with unpleasant odour. B.p. 240°.

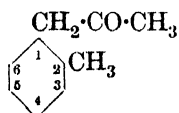
*Amide*: plates from  $H_2O$ . M.p. 185°. Sol. hot EtOH. Spar. sol. cold  $H_2O$ ,  $Et_2O$ . Sublimes.

*Nitrile*: *p*-tolubenzyl cyanide, *p*-xylyl cyanide. F.p. 18°. M.p. 18°. B.p. 242–3°.  $D_{25}^{20}$  0.9922.

Schorigin, *Ber.*, 1910, **43**, 1941.

Radziszewski, Wispek, *Ber.*, 1885, **18**, 1281.

**o-Tolylacetone** ( $\beta$ -Keto- $\alpha$ -*o*-tolylpropane, 2-acetonyltoluene, methyl *o*-tolubenzyl ketone, methyl *o*-xylyl ketone)



$C_{10}H_{12}O$  MW, 148

Liq. B.p. 227°, 122°/23 mm.

*Oxime*: m.p. 75°.

*Semicarbazone*: m.p. 181°.

Ruzicka, Ehmann, Weisz, *Helv. Chim. Acta*, 1932, **15**, 159.

Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 195.

**m-Tolylacetone** ( $\beta$ -Keto- $\alpha$ -*m*-tolylpropane, 3-acetonyltoluene, methyl *m*-tolubenzyl ketone, methyl *m*-xylyl ketone).

Liq. B.p. 228–9°.  $D_0^{20}$  1.019.

*Semicarbazone*: m.p. 139°.

See last reference above.

**p-Tolylacetone** ( $\beta$ -Keto- $\alpha$ -*p*-tolylpropane, 4-acetonyltoluene, methyl *p*-tolubenzyl ketone, methyl *p*-xylyl ketone).

Liq. B.p. 232–3°, 109–10°/12 mm. Forms bisulphite comp.

Dict. of Org. Comp.—III.

*Oxime*: prisms from pet. ether. M.p. 90–1°. *Semicarbazone*: m.p. 158°.

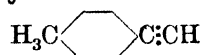
Ruzicka, Ehmann, Rierink, *Helv. Chim. Acta*, 1932, **15**, 160.

Tiffeneau, *Ann. chim. phys.*, 1907, **10**, 195.

$\omega$ -**p-Tolylacetophenone**.

See 4-Methyldeoxybenzoin.

**p-Tolylacetylene**



$C_9H_8$  MW, 116

Prisms. M.p. 23°. B.p. 168°, 65–7°/18 mm.  $D_{25}^{20}$  0.9159.  $n_D^{25}$  1.5447. Gives explosive Cu and Ag derivs.

Otto, *J. Am. Chem. Soc.*, 1934, **56**, 1393.

Vaughn, *ibid.*, 2064.

Gattermann, *Ann.*, 1906, **347**, 359.

**2-p-Tolylacrolein**.

See *p*-Methylcinnamaldehyde.

**2-Tolylacrylic Acid**.

See *o*-, *m*-, and *p*-Methylcinnamic Acid.

**Tolylaminophenol**.

See Hydroxy-methyldiphenylamine.

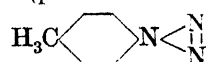
**Tolyanisidine**.

See under Hydroxy-methyldiphenylamine.

**N-Tolylanthranilic Acid**.

See Methyldiphenylamine-carboxylic Acid.

**p-Tolyl azide** (*p*-Azidotoluene)



$C_7H_7N_3$  MW, 133

Yellow oil. B.p. 80°/10 mm. Decomp. at 180°.  $D_4^{25}$  1.0527. Volatile in steam.

Lindemann, Thiele, *Ber.*, 1928, **61**, 1529.

Ponzio, *Gazz. chim. ital.*, 1916, **46**, 57.

Dimroth, Pfister, *Ber.*, 1910, **43**, 2760.

**Tolylazoimide**.

See Methylbenztriazole.

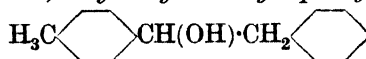
**Tolylbenzoic Acid**.

See Methyldiphenyl-carboxylic Acid.

**Tolylbenzylamine**.

See *N*-Benzyltoluidine.

**p-Tolylbenzylcarbinol** ( $\beta$ -Phenyl- $\alpha$ -*p*-tolylethyl alcohol,  $\alpha$ -hydroxy-4-methyldiphenylethane)



$C_{15}H_{16}O$  MW, 212

Needles. M.p. 107–8° (66°). Dist. without decomp. above 360°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

Tiffeneau, Lévy, *Bull. soc. chim.*, 1931, **49**, 1738.

Mann, *Ber.*, 1881, **14**, 1646.

**Tolyl benzyl Ketone.**

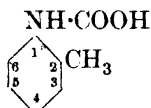
See 2', 3', and 4'-Methyldeoxybenzoin.

**Tolylbutane.**

See Butyltoluene.

**Tolylbutenone.**

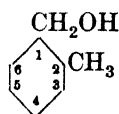
See Methyl methylstyryl Ketone.

**o-Tolylcarbamic Acid** (o-Methylcarbanilic acid) $\text{C}_8\text{H}_9\text{O}_2\text{N}$ 

MW, 151

 $\beta$ -Chloroethyl ester :  $\text{C}_{10}\text{H}_{12}\text{O}_2\text{NCl}$ . MW, 213.5. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $45^\circ$ . B.p.  $209-10^\circ/37$  mm. $\gamma$ -Chloropropyl ester :  $\text{C}_{11}\text{H}_{14}\text{O}_2\text{NCl}$ . MW, 227.5. Needles from ligroin. M.p.  $49^\circ$  ( $46-46.5^\circ$ ). B.p.  $182.5^\circ/4.5$  mm. ( $170-5^\circ/5$  mm.).Butyl ester :  $\text{C}_{12}\text{H}_{17}\text{O}_2\text{N}$ . MW, 207. Prisms. M.p.  $45.5^\circ$ .Chattaway, Saerens, *J. Chem. Soc.*, 1920, 117, 711.Adams, Segur, *J. Am. Chem. Soc.*, 1923, 45, 787.Adams, Pierce, *ibid.*, 793.**p-Tolylcarbamic Acid** (p-Methylcarbanilic acid).Et ester : p-tolylurethane.  $\text{C}_{10}\text{H}_{13}\text{O}_2\text{N}$ . MW, 179. B.p.  $243-7^\circ$ ,  $128-32^\circ/12$  mm.Butyl ester : prisms. M.p.  $63^\circ$ . $\beta$ -Chloroethyl ester : cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $61^\circ$ . $\gamma$ -Chloropropyl ester : straw-coloured oil. B.p.  $188^\circ/4.5$  mm.  $D_{20}^{20}$  1.186.  $n_D^{18}$  1.494.

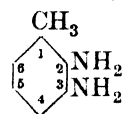
See previous references.

**o-Tolylcarbinol** (2-Methylbenzyl alcohol) $\text{C}_8\text{H}_{10}\text{O}$ 

MW, 122

Needles. M.p.  $36^\circ$ . B.p.  $219^\circ$ ,  $135^\circ/30$  mm.  $D_4^{20}$  1.023. Sol. 100 parts cold  $\text{H}_2\text{O}$ , 60 parts at the boil. Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Ox.  $\rightarrow$  o-toluic aldehyde.Me ether :  $\text{C}_9\text{H}_{12}\text{O}$ . MW, 136. Oil with unpleasant odour. B.p.  $187-8^\circ$ .Et ether :  $\text{C}_{10}\text{H}_{14}\text{O}$ . MW, 150. Sweet-smelling oil. B.p.  $208-10^\circ$ .Isoamyl ether :  $\text{C}_{13}\text{H}_{20}\text{O}$ . MW, 192. Oil with aromatic odour. B.p.  $125^\circ/15$  mm.Acetyl : liq. B.p.  $228-30^\circ/753$  mm.Urethane : m.p.  $79^\circ$ .Colson, *Ann. chim.*, 1885, 6, 115.**m-Tolylcarbinol** (3-Methylbenzyl alcohol).Oil. B.p.  $217^\circ$ .  $D^{17}$  0.9157. Sol. 20 parts cold  $\text{H}_2\text{O}$ . Misc. with EtOH,  $\text{Et}_2\text{O}$ . Ox.  $\rightarrow$  m-toluic aldehyde + m-toluic acid.Et ether : b.p.  $202^\circ/740$  mm.  $D^{17}$  0.9302.Acetyl : oil. B.p.  $226^\circ$ .Colson, *Ann. chim.*, 1885, 6, 117.Radziszewski, Wispek, *Ber.*, 1882, 15, 1747.**p-Tolylcarbinol** (4-Methylbenzyl alcohol).Needles. M.p.  $60^\circ$ . B.p.  $217^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. cold  $\text{H}_2\text{O}$ , mod. sol. hot.Et ether : b.p.  $203^\circ/740$  mm.  $D^{17}$  0.9304.Acetyl : oil. B.p.  $220^\circ$ .Oddo, *Gazz. chim. ital.*, 1911, 41, 285.Cannizzaro, *Ann.*, 1862, 124, 255.**2-p-Tolylcinchoninic Acid.**

See 2-p-Tolylquinoline-4-carboxylic Acid.

**2:3-Tolylenediamine** (2:3-Diaminotoluene, 3-methyl-o-phenylenediamine) $\text{C}_7\text{H}_{10}\text{N}_2$ 

MW, 122

Cryst. M.p.  $63-4^\circ$ . B.p.  $255^\circ$ . Sol. most. org. solvents. Boiling  $\text{H}\cdot\text{COOH} \rightarrow$  4-methylbenziminazole.Gabriel, Thieme, *Ber.*, 1919, 52, 1081.**2:4-Tolylenediamine** (2:4-Diaminotoluene, 4-methyl-m-phenylenediamine, unsym.-m-toluylenediamine).Needles from  $\text{H}_2\text{O}$ , prisms from EtOH. M.p.  $99^\circ$ . B.p.  $292^\circ$ ,  $148-50^\circ/8$  mm. Very sol. boiling  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Aq. sol. darkens in air. Reduces warm  $\text{NH}_3\cdot\text{AgNO}_3$ . Ox.  $\rightarrow$  2-amino-4-nitrotoluene + 2:4-dinitrotoluene. Sol. in dil. HCl on warming with dil.  $\text{KClO}_3 \rightarrow$  light violet col.2-N-Me :  $\text{C}_8\text{H}_{12}\text{N}_2$ . MW, 136. Oil. B.p.  $273^\circ$ . 2-N-Benzoyl : needles. M.p.  $167^\circ$ .

2-N-Di-Me : see 2-N-Dimethyl-2:4-tolylenediamine.

4-N-Di-Me : see 4-N-Dimethyl-2:4-tolylenediamine.

N-Tetra-Me :  $\text{C}_{11}\text{H}_{18}\text{N}_2$ . MW, 178. Brownish-yellow oil. B.p.  $255-6^\circ/757$  mm.,  $148-50^\circ/24-6$  mm.  $D^{24}$  0.9661. Picrate : prisms from AcOEt. M.p.  $162-3^\circ$ .

2-N-Et :  $C_9H_{14}N_2$ . MW, 150. Liq. B.p. 274-5°.

4-N-Et : liq. B.p. 289-91°.

2-N-Di-Et :  $C_{11}H_{18}N_2$ . MW, 178. Yellow oil. B.p. 259°. Volatile in steam.  $B, 2HCl$  : cryst. +  $H_2O$ . M.p. 213-15°.

4-N-Butyl :  $C_{11}H_{18}N_2$ . MW, 178. Needles from  $Et_2O$ . M.p. 53°. Spar. sol.  $H_2O$ , ligroin.

4-N-Phenyl : see 3-Amino-4-methyldiphenylamine.

4-N-Et-4-Phenyl :  $C_{15}H_{18}N_2$ . MW, 226. Cryst. from ligroin. M.p. 59-60°.

4-N-p-Tolyl : see 3-Amino-4 : 4'-dimethyldiphenylamine.

2-N-Benzyl :  $C_{14}H_{18}N_2$ . MW, 212. Needles. M.p. 81°. Very sol.  $EtOH$ ,  $Et_2O$ . Insol. cold  $H_2O$ .

4-N-Formyl : cryst. from  $H_2O$ . M.p. 113-14°.

2 : 4-N-Diformyl : needles from  $H_2O$ . M.p. 176-7°.

2-N-Acetyl : needles. M.p. 140°.

4-N-Acetyl : prisms or needles. M.p. 161-5°.

2 : 4-N-Diacetyl : needles. M.p. 224°.

4-N-Benzoyl : prisms. M.p. 142°.

2 : 4-N-Dibenzoyl : plates from  $AcOH$ . M.p. 224°.

2-N-Benzenesulphonyl : plates from  $EtOH$ . M.p. 138°.

4-N-Benzenesulphonyl : m.p. 138°.

2 : 4-N-Dibenzenesulphonyl : needles. M.p. 191°.

4-N-p-Toluenesulphonyl : cryst. powder. M.p. 160°.

2 : 4-N-Di-p-toluenesulphonyl : needles. M.p. 192-3°.

Mahood, Schaffner, *Organic Syntheses*, 1931, XI, 32.

Gnehm, Blumer, *Ann.*, 1899, 304, 106.

**2 : 5-Tolylenediamine** (2:5-Diaminotoluene, 2-methyl-p-phenylenediamine, p-toluylenediamine).

Plates from  $C_6H_6$ . M.p. 64°. B.p. 273-4°. Sol.  $H_2O$ ,  $EtOH$ ,  $Et_2O$ , hot  $C_6H_6$ . Spar. sol. cold  $C_6H_6$ .  $MnO_2 + H_2SO_4 \rightarrow$  toluquinone.

2-N-Me :  $C_8H_{12}N_2$ . MW, 136. Thick oil. B.p. 276-276.5°. Rapidly oxidises in air. 2-p-Toluenesulphonyl : plates from  $EtOH.Aq$ . M.p. 118-19°.

2-N-Di-Me : see 2-N-Dimethyl-2 : 5-tolylenediamine.

5-N-Di-Me : see 5-N-Dimethyl-2 : 5-tolylenediamine.

2 : 5-N-Tetra-Me :  $C_{11}H_{18}N_2$ . MW, 178. Oil. B.p. 260°.  $FeCl_3 \rightarrow$  blue col. Monomethiodide : needles from  $H_2O$ . M.p. 160°.

2-N-Et :  $C_9H_{14}N_2$ . MW, 150. Thick oil. B.p. 272° (in hydrogen).  $B, 2HCl$  : m.p. 124° decomp. 5-N-Benzoyl : needles from  $C_6H_6$ -ligroin. M.p. 174°.

2-N-Di-Et :  $C_{11}H_{18}N_2$ . MW, 178. M.p. 24°. B.p. 266-7°.

5-N-o-Tolyl : see 4-Amino-3 : 2'-dimethyldiphenylamine.

2 : 5-N-Di-p-tolyl :  $C_{21}H_{22}N_2$ . MW, 302. Plates from  $AcOH$ . M.p. 112-13°.

2 : 5-N-Diacetyl : prisms from  $EtOH.Aq$ . M.p. 220°.

2-N-Benzenesulphonyl : needles from  $H_2O$ . M.p. 147°.

2-N-p-Toluenesulphonyl : prisms from  $EtOH.Aq$ . M.p. 150°.

Nietzki, *Ber.*, 1877, 10, 1157.

**2 : 6-Tolylenediamine** (2:6-Diaminotoluene, 2-methyl-m-phenylenediamine).

Prisms from  $H_2O$ . M.p. 105°.  $FeCl_3 \rightarrow$  deep brown col.

N-Diacetyl : m.p. 202-3°.

Ullmann, *Ber.*, 1884, 17, 1959.

**3 : 4-Tolylenediamine** (3:4-Diaminotoluene, 4-methyl-o-phenylenediamine, unsym.-o-toluylenediamine).

Plates from ligroin. M.p. 89-90°. B.p. 265°. Mod. sol. cold  $H_2O$ . Sublimes. Base and salts rapidly oxidise in aq. sol. in air. Boiling  $H.COOH \rightarrow$  5-methylbenzimidazole.

4-N-Me :  $C_8H_{12}N_2$ . MW, 136. Plates from  $Et_2O$ . M.p. 43-4°. B.p. 260°/752 mm.  $B, HCl$  : plates from  $EtOH$ . M.p. 175-80°.  $B, 2HCl$  : prisms from  $EtOH$ . M.p. 175-85° decomp.  $(B.COOH)_2$  : needles. M.p. 124°. Picrate : yellow cryst. M.p. 164°. 3 : 4-N-Diacetyl : plates from  $H_2O$ . M.p. 183-4°. 4-N-p-Toluenesulphonyl : needles. M.p. 133°.

3 : 4-N-Di-Me :  $C_9H_{14}N_2$ . MW, 150. Oil. B.p. 259-60°/740 mm.  $FeCl_3 \rightarrow$  red col.  $B, 2HCl$  : needles. M.p. 125°.

4-N-Di-Me : see 4-N-Dimethyl-3 : 4-tolylenediamine.

3 : 4-N-Tetra-Me :  $C_{11}H_{18}N_2$ . MW, 178. Liq. B.p. 224-5-225-5°/717 mm.

3-N-Et :  $C_9H_{14}N_2$ . MW, 150. Needles from pet. ether. M.p. 59°. Becomes violet in air.  $FeCl_3 \rightarrow$  red col.

4-N-Et : plates from  $CS_2.Aq$ . M.p. 55°. Very sol. most org. solvents. Not very stable.  $B, HCl$  : cryst. from  $EtOH$ . M.p. 176°.  $B_2(COOH)_2$  : needles. M.p. 151°. 3-N-Acetyl : cryst. from  $EtOH$ . M.p. 177°.

3 : 4-N-Di-Et : *see sym.*-Diethyl-3 : 4-tolylene-diamine.

4-N-Butyl :  $C_{11}H_{18}N_2$ . MW, 178. Very readily oxidises. 4-N-Acetyl : needles from  $Et_2O$ -pet. ether. M.p.  $102^\circ$ . 3 : 4-N-Diacetyl : cryst. from  $EtOH.Aq.$  M.p.  $130^\circ$ .

3-N-Phenyl : *see* 6-Amino-3-methyldiphenylamine.

4-N-Phenyl : *see* 2-Amino-4-methyldiphenylamine.

3-N-p-Tolyl : *see* 6-Amino-3 : 4'-dimethyldiphenylamine.

4-N-p-Tolyl : *see* 2-Amino-4 : 4'-dimethyldiphenylamine.

3-N-Acetyl : cryst. from  $CHCl_3$ -pet. ether. M.p. about  $95^\circ$ .

4-N-Acetyl : needles. M.p.  $131-2^\circ$ .

3 : 4-N-Diacetyl : needles from  $H_2O$ . M.p.  $210^\circ$ .

3-N-Benzoyl : needles from  $C_6H_6$ . M.p.  $158^\circ$ .

4-N-Benzoyl : needles from  $EtOH$  or  $C_6H_6$ . M.p.  $193-4^\circ$ .

3 : 4-N-Dibenzoyl : needles from  $AcOH$ . M.p.  $263-4^\circ$ .

3-N-Benzenesulphonyl : needles from  $C_6H_6$ . M.p.  $134-5^\circ$ .

4-N-Benzenesulphonyl : needles from  $EtOH.Aq.$  M.p.  $146-7^\circ$ .

3 : 4-N-Dibenzenesulphonyl : plates from  $EtOH$ . M.p.  $178-9^\circ$ .

4-N-p-Toluenesulphonyl : needles. M.p.  $140^\circ$ .

Reilly, Hickinbottom, *J. Chem. Soc.*, 1919, 115, 177.

Fischer, *Ber.*, 1893, 26, 194.

Bamberger, Wulz, *Ber.*, 1891, 24, 2082.

Noelting, Stoecklin, *ibid.*, 565.

**3 : 5-Tolylenediamine** (3 : 5-Diaminotoluene, 5-methyl-m-phenylenediamine, sym.-m-toluylenediamine).

Oil. B.p.  $283-5^\circ$ . Very sol.  $H_2O$ .

$B_2HCl$  : needles. M.p.  $255-60^\circ$  decomp.

3 : 5-N-Diphenyl :  $C_{19}H_{18}N_2$ . MW, 274. Cryst. from  $AcOH$ . M.p.  $105^\circ$ . Sol. cold  $EtOH$ ,  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ .

3 : 5-N-Diacetyl : needles. M.p.  $160^\circ$ . 3 : 5-N-Dibenzoyl : needles from  $C_6H_6$ -ligroin. M.p.  $190-1^\circ$ .

3 : 5-N-Diacetyl : prisms from  $EtOH$ . M.p.  $235-6^\circ$ .

Davis, *J. Chem. Soc.*, 1902, 81, 873.

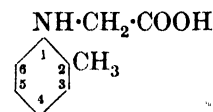
**1-Tolylolethyl Alcohol.**

*See* Methyltolylcarbinol.

**Tolyloethylene.**

*See* Methylstyrene.

**o-Tolylglycine** (o-Toluidinoacetic acid)



$C_9H_{11}O_2N$

MW, 165

Needles from  $EtOH$ . M.p.  $149-50^\circ$  ( $160^\circ$ ). Very sol.  $EtOH$ ,  $Et_2O$ . Insol. cold  $H_2O$ .  $k = 5.9 \times 10^{-5}$  at  $25^\circ$ . Reduces silver salts.

*Et ester* :  $C_{11}H_{15}O_2N$ . MW, 193. Cryst. M.p.  $26^\circ$ . B.p.  $280^\circ$ .  $D^{20} 1.058$ .

*Amide* :  $C_9H_{12}ON_2$ . MW, 164. Needles from  $H_2O$ . M.p.  $140^\circ$ . Sol.  $EtOH$ , hot  $H_2O$ .

*N-Et* :  $C_{11}H_{15}O_2N$ . MW, 193. Cryst. from  $C_6H_6$ . M.p.  $63-4^\circ$ .

*N-Formyl* : cryst. from  $H_2O$ . M.p.  $113-15^\circ$ .

*N-Acetyl* : plates from  $EtOH.Aq.$  M.p.  $210-12^\circ$ .  $k = 2.19 \times 10^{-4}$  at  $25^\circ$ .

*N-Chloroacetyl* : plates from  $C_6H_6$ . M.p.  $116-17^\circ$ .

*N-Bromoacetyl* : plates from  $H_2O$ . M.p.  $124^\circ$ .

Staats, *Ber.*, 1880, 13, 137.

Ehrlich, *Ber.*, 1883, 16, 204.

Steppes, *J. prakt. Chem.*, 1900, 62, 491.

**m-Tolylglycine** (m-Toluidinoacetic acid).

*Et ester* : plates from  $EtOH$ . M.p.  $68^\circ$ . Sol.  $EtOH$ ,  $Et_2O$ ,  $AcOH$ . Very spar. sol. hot  $H_2O$ .

Ehrlich, *Ber.*, 1882, 15, 2011.

Gault, *Bull. soc. chim.*, 1908, 3, 372.

**p-Tolylglycine** (p-Toluidinoacetic acid).

Plates from  $Et_2O$ -pet. ether. M.p.  $132^\circ$  ( $120-1^\circ$ ). Insol. pet. ether, cold  $H_2O$ . Unstable in air.  $k = 1.5 \times 10^{-5}$  at  $25^\circ$ . Reduces Tollen's reagent.

*Et ester* : m.p.  $52-3^\circ$  ( $48-9^\circ$ ). B.p.  $279^\circ$  slight decomp. Very sol.  $Et_2O$ . Mod. sol. cold  $EtOH$ . Very spar. sol. hot  $H_2O$ .

*Amide* : cryst. from  $H_2O$ . M.p.  $168^\circ$  ( $162-3^\circ$ ). Sol.  $EtOH$ ,  $C_6H_6$ , hot  $H_2O$ . Insol. cold  $H_2O$ .

*Nitrile* :  $C_9H_{10}N_2$ . MW, 146. Cryst. M.p.  $62^\circ$ . Very sol. common org. solvents except ligroin. *N-Me* : m.p.  $57^\circ$ . B.p.  $156-7^\circ/9 \text{ mm.}$

*Anilide* : needles from  $H_2O$ . M.p.  $82-3^\circ$ .

*N-Acetyl* : plates from  $H_2O$ . M.p.  $175-6^\circ$ .

Steppes, *J. prakt. Chem.*, 1900, 62, 487.

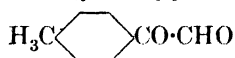
Bischoff, Hausdörfer, *Ber.*, 1892, 25, 2282.

v. Miller, Plöchl, Sieber, *Ber.*, 1898, 31, 2715.

M.L.B., D.R.P., 175,797, (*Chem. Zentr.*, 1906, II, 1700).

**Tolylglycollic Acid.**

*See* Methylmandelic Acid.

**p-Tolylglyoxal** (*p-Toluyaldehyde*) $C_9H_8O_2$ 

MW, 148

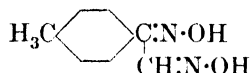
Needles +  $1H_2O$ . M.p. anhyd. 111–12° (101°).  
Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol.  
H<sub>2</sub>O, ligroin.

*Aldoxime*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 100°.  
*Acetyl*: plates from MeOH. M.p. 67–8°.  
*Phenylhydrazone*: plates from EtOH. M.p. 165°.

*Dioxime*: see *p-Tolylglyoxime*.

*Phenylosazone*: yellow needles from EtOH.  
M.p. 145°.

Müller, v. Pechmann, *Ber.*, 1889, **22**, 2560.  
Neuberg, Ostendorf, *Biochem. Z.*, 1935,  
**279**, 459.

**p-Tolylglyoxime** $C_9H_{10}O_2N_2$ 

MW, 178

*α-Form*:

M.p. 170–1°. Above m.p. → *β-form*.

*Diacetyl*: m.p. 115°.

*Monobenzyl*: m.p. 147–8°.

*β-Form*:

M.p. 192–3°. Forms Ni salt.

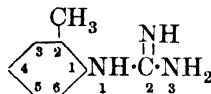
*Diacetyl*: m.p. 73–4°.

*Dibenzoyl*: m.p. 170°.

Avogadro, *Gazz. chim. ital.*, 1923, **53**, 698.

**Tolylglyoxylic Acid.**

See Toluyalformic Acid.

**o-Tolylguanidine** (*Guanyl-o-toluidine*) $C_8H_{11}N_3$ 

MW, 149

*B,HCl*: cryst. from Me<sub>2</sub>CO–Et<sub>2</sub>O. M.p. 133–  
5°. Sol. H<sub>2</sub>O, Me<sub>2</sub>CO. Insol. Et<sub>2</sub>O.

*Nitrate*: cryst. from H<sub>2</sub>O. M.p. 133°. Very  
bitter taste.

*Picrate*: m.p. 223–4°.

*3-Phenyl*: see Phenyl-*o*-tolylguanidine.

Braun, *J. Am. Chem. Soc.*, 1933, **55**, 1281.

M.L.B., D.R.P., 172,979, (*Chem. Zentr.*,  
1906, II, 984).

**m-Tolylguanidine** (*Guanyl-m-toluidine*).

*Sulphate*: cryst. from EtOH–Et<sub>2</sub>O. M.p.  
215–17°. Sol. H<sub>2</sub>O. Insol. Et<sub>2</sub>O.

Braun, *J. Am. Chem. Soc.*, 1933, **55**, 1282.

**p-Tolylguanidine** (*Guanyl-p-toluidine*).

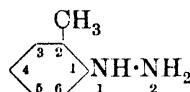
*B,HCl*: cryst. from EtOH–Et<sub>2</sub>O. M.p.  
136–7°. Sol. H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O.

*B,HNO<sub>3</sub>*: m.p. 146–7°. Very bitter taste.

*3-Phenyl*: see Phenyl-*p*-tolylguanidine.

See previous reference and also

Kämpf, *Ber.*, 1904, **37**, 1683.

**o-Tolylhydrazine** (*o-Hydrazinotoluene*) $C_7H_{10}N_2$ 

MW, 122

Needles. M.p. 59° (56°). Sol. EtOH, Et<sub>2</sub>O,  
CHCl<sub>3</sub>. Spar. sol. cold ligroin.

*B,HNO<sub>3</sub>*: m.p. 98–100° (sinters about 75°).

*2-N-Phenyl*: see 2-Methylhydrazobenzene.

*2-N-Formyl*: plates from H<sub>2</sub>O. M.p. 120°.

*2-N-Acetyl*: leaflets from H<sub>2</sub>O. M.p. 104°.

*2-N-Propionyl*: plates from H<sub>2</sub>O. M.p.  
83–4°.

*2-N-Isobutyryl*: leaflets from C<sub>6</sub>H<sub>6</sub>–pet. ether.  
M.p. 93°.

*2-N-Benzoyl*: needles. M.p. 180°.

Fischer, Bösler, *Ann.*, 1882, **212**, 338.

Gallinek, v. Richter, *Ber.*, 1885, **18**, 3175.

**m-Tolylhydrazine** (*m-Hydrazinotoluene*).

Oil. B.p. 240–4°.

*B,HNO<sub>3</sub>*: needles. M.p. 145–7°.

*2-N-Phenyl*: see 3-Methylhydrazobenzene.

*2-N-Propionyl*: m.p. 131°.

Buchka, Schachtebeck, *Ber.*, 1889, **22**,  
841.

**p-Tolylhydrazine** (*p-Hydrazinotoluene*).

Leaflets from Et<sub>2</sub>O. M.p. 65–6° (61°). B.p.  
240–4° slight decomp. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.  
Spar. sol. H<sub>2</sub>O.

*B,HNO<sub>3</sub>*: leaflets. M.p. 152–3°.

*2-N-Phenyl*: see 4-Methylhydrazobenzene.

*2-N-Formyl*: prisms or needles from EtOH.  
M.p. 166–5°.

*2-N-Acetyl*: cryst. M.p. 130° (127°).

*2-N-Propionyl*: needles from H<sub>2</sub>O. M.p. 170°.

*2-N-Isobutyryl*: leaflets. M.p. 147–8°.

*1-N-Benzoyl*: leaflets from C<sub>6</sub>H<sub>6</sub>. M.p.  
68–70°. *2-N-Acetyl*: needles from C<sub>6</sub>H<sub>6</sub>. M.p.  
135°.

*2-N-Benzoyl*: leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 146°.  
*1:2-N-Dibenzoyl*: leaflets from EtOH. M.p.  
188°.

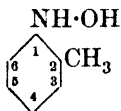
McPherson, Stratton, *J. Am. Chem. Soc.*,  
1915, **37**, 908.

Bamberger, *Ber.*, 1898, **31**, 582.

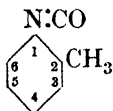
Fischer, *Ber.*, 1876, **9**, 890.

**Tolyl  $\alpha$ -hydroxybenzyl Ketone.**

See Methylbenzoin.

**N-*o*-Tolyldihydroxylamine** (*o*-Hydroxyl-aminotoluene) $C_7H_9ON$ 

MW, 123

Needles from  $Et_2O-C_6H_6$ . M.p.  $44^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. ligroin.Bamberger, Rising, *Ann.*, 1901, **316**, 278.  
Bretschneider, *J. prakt. Chem.*, 1897, **55**, 293.**N-*m*-Tolyldihydroxylamine** (*m*-Hydroxyl-aminotoluene).Leaflets from  $C_6H_6$ -pet. ether. M.p.  $68.5^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , hot  $C_6H_6$ . Mod. sol. hot  $H_2O$ . Spar. sol. ligroin.Bamberger, Rising, *Ann.*, 1901, **316**, 283.**N-*p*-Tolyldihydroxylamine** (*p*-Hydroxyl-aminotoluene).Leaflets from  $C_6H_6$ . M.p.  $94^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , hot  $C_6H_6$ . Insol. cold ligroin. At  $115-20^\circ \rightarrow$  *p*-azoxytoluene.Willstätter, Kubli, *Ber.*, 1908, **41**, 1937.Bamberger, Rising, *Ann.*, 1901, **316**, 280.**Tolylisobutylamine.**See *N*-Isobutyl-toluidine.***o*-Tolyl isocyanate** (*o*-Tolylcarbonimide) $C_8H_7ON$ 

MW, 133

Liq. B.p.  $185-6^\circ$ .Gattermann, Cantzler, *Ber.*, 1892, **25**, 1086.Haager, Doht, *Monatsh.*, 1906, **27**, 271.***m*-Tolyl isocyanate** (*m*-Tolylcarbonimide).B.p.  $195-8^\circ$  ( $183^\circ$ ). *m*-Toluidine  $\rightarrow$  di-*m*-tolylurea.Haager, Doht, *Monatsh.*, 1906, **27**, 273.Gattermann, Cantzler, *Ber.*, 1892, **25**, 1089.***p*-Tolyl isocyanate** (*p*-Tolylcarbonimide).Liq. B.p.  $187^\circ/751$  mm.

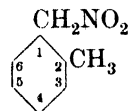
See first reference above and also

Kühn, Henschel, *Ber.*, 1888, **21**, 505  
(Note).**Tolynaphthylamine.**

See under Naphthylamine.

**Tolyl naphthyl sulphide.**

See under Thionaphthol.

***o*-Tolynitromethane** ( $\omega$ -Nitro-*o*-xylene) $C_8H_9O_2N$ 

MW, 151

M.p.  $12-14^\circ$ . B.p.  $145-6^\circ/23$  mm., slight decomp.,  $138-9^\circ/20$  mm.  $D_4^{18}$  1.1423.  $n_D^{18}$  1.5439. Turns red on standing.Konowaloff, *Chem. Zentr.*, 1905, **II**, 817.Wislicenus, Wren, *Ber.*, 1905, **38**, 503.***m*-Tolynitromethane** ( $\omega$ -Nitro-*m*-xylene).Yellowish liq. B.p.  $140^\circ/35$  mm. decomp.,  $128-32^\circ/19$  mm.  $D_4^0$  1.1370.Konowaloff, *Chem. Zentr.*, 1899, **I**, 1238.Heilmann, *Ber.*, 1890, **23**, 3165.Wislicenus, Wren, *Ber.*, 1905, **38**, 505.***p*-Tolynitromethane** ( $\omega$ -Nitro-*p*-xylene).M.p.  $11-12^\circ$ . B.p.  $150-1^\circ/35$  mm. slight decomp.  $D_4^0$  1.1234.  $n_D^{20}$  1.53106.Konowaloff, *Chem. Zentr.*, 1899, **I**, 1238.Wislicenus, Wren, *Ber.*, 1905, **38**, 506.**Tolylphenetidine.**

See under Hydroxy-methyldiphenylamine.

**4-*p*-Tolylphenol.**

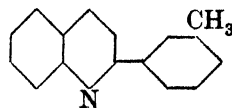
See 4'-Hydroxy-4-methyldiphenyl.

**Tolylphenylenediamine.**

See 4'-Amino-2-methyldiphenylamine, 2'-Amino-4-methyldiphenylamine, and 4'-Amino-4-methyldiphenylamine.

**2-Tolylpropionaldehyde.**See *o*-, and *p*-Methylhydrocinnamaldehyde.**1-*p*-Tolylpropionic Acid.**See *p*-Methylhydratropic Acid.**2-Tolylpropionic Acid.**See *o*-, *m*-, and *p*-Methylhydrocinnamic Acid.**1-Tolyl-2-pyridylethylene.**

See Methylstyrylpyridine.

**2-*m*-Tolylquinoline** ( $\psi$ -Flavoline) $C_{16}H_{13}N$ 

MW, 219

Needles from  $C_6H_6$ -ligroin. M.p.  $77^\circ$ .Weidel, Bamberger, *Monatsh.*, 1888, **9**, 109.

**2-*p*-Tolylquinoline.**

Pale yellow plates from EtOH.Aq. M.p. 83°. B.p. 240°/15 mm.

*Picrate*: m.p. 194°.

*Methiodide*: decomp. at 192°.

*Methosulphate*: needles from EtOH-Et<sub>2</sub>O. M.p. 158-9°.

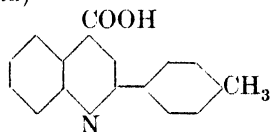
*Methopicrate*: m.p. 157-8°.

Le Fèvre, Le Fèvre, Pearson, *J. Chem. Soc.*, 1934, 41.

v. Braun, Brauns, *Ber.*, 1927, 60, 1255.

**2-*p*-Tolylquinoline-4-carboxylic Acid (4'-**

*Methylcinchophene*, 4'-*methylatophan*, 2-*p*-tolyl-*cinchonic acid*)



C<sub>17</sub>H<sub>13</sub>O<sub>2</sub>N

MW, 263

Cryst. from EtOH. M.p. 211°. Loses CO<sub>2</sub> at 250°.

*Me ester*: C<sub>18</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 277. Cryst. M.p. 101°.

*Et ester*: C<sub>19</sub>H<sub>17</sub>O<sub>2</sub>N. MW, 291. Yellow needles from 70% EtOH. M.p. 54°. Sol. most org. solvents.

*Propyl ester*: C<sub>20</sub>H<sub>19</sub>O<sub>2</sub>N. MW, 305. Yellow cryst. from EtO<sub>2</sub>. M.p. 32°. Sol. usual solvents.

*2-Chloroethyl ester*: C<sub>19</sub>H<sub>16</sub>O<sub>2</sub>NCl. MW, 325.5. Needles from 70% MeOH. M.p. 79°.

*Chloride*: C<sub>17</sub>H<sub>12</sub>ONCl. MW, 281.5. *B,HCl*: cryst. M.p. 188°.

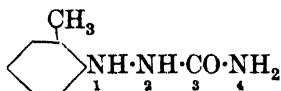
*Amide*: C<sub>17</sub>H<sub>14</sub>ON<sub>2</sub>. MW, 262. Needles from EtOH. M.p. 208°. Sol. cold Me<sub>2</sub>CO. Less sol. C<sub>6</sub>H<sub>6</sub>, toluene.

*Hydrazide*: cryst. from chlorobenzene. M.p. 232-3°. Sol. propyl alcohol, amyl alcohol, chlorobenzene. Loss sol. MeOH, EtOH, isopropyl alcohol, C<sub>6</sub>H<sub>6</sub>, toluene, xylene. Insol. Et<sub>2</sub>O. *Picrate*: prisms from EtOH. Does not melt below 300°.

v. Braun, Brauns, *Ber.*, 1927, 60, 1255.

John, Ottawa, *J. prakt. Chem.*, 1931, 131, 314.

Du Puis, Lindwall, *J. Am. Chem. Soc.*, 1934, 56, 471.

**1-*o*-Tolylsemicarbazide**

C<sub>8</sub>H<sub>11</sub>ON<sub>3</sub>

MW, 165

Needles. M.p. 159-60°. Mod. sol. H<sub>2</sub>O. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

4-*N-Me*: m.p. 158-9°.

4-*N-Et*: needles from EtOH. M.p. 130-1°.

4-*N-Phenyl*: needles from EtOH. M.p. 142°.

Pinner, *Ber.*, 1888, 21, 1221.

**1-*m*-Tolylsemicarbazide.**

Leaflets from H<sub>2</sub>O or EtOH.Aq. M.p. 183-4°. Insol. Et<sub>2</sub>O. Employed as antipyretic under name of Marietin.

4-*N-Phenyl*: needles from EtOH. M.p. 159°.

Bayer, D.R.P., 157,572, (*Chem. Zentr.*, 1905, I, 196).

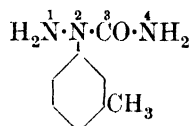
**1-*p*-Tolylsemicarbazide.**

Leaflets or needles. M.p. 190-1° (187-8°). Sol. hot H<sub>2</sub>O, hot EtOH.

4-*N-Phenyl*: needles from AcOEt. M.p. 171°.

Bamberger, *Ber.*, 1902, 35, 1428.

Pinner, *Ber.*, 1888, 21, 1222.

**2-*m*-Tolylsemicarbazide**

C<sub>8</sub>H<sub>11</sub>ON<sub>3</sub>

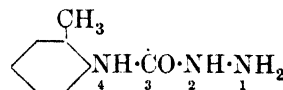
MW, 165

Needles. M.p. 88°. At 140° → 1-*m*-tolylsemicarbazide.

4-*N-Me*: m.p. 119-20°.

4-*N-Phenyl*: needles from EtOH-Et<sub>2</sub>O. M.p. 112°.

Bayer, D.R.P., 163,035, (*Chem. Zentr.*, 1905, II, 1298).

**4-*o*-Tolylsemicarbazide**

C<sub>8</sub>H<sub>11</sub>ON<sub>3</sub>

MW, 165

Cryst. from H<sub>2</sub>O or EtOH.Aq. M.p. 142-3° decomp.

*B,HCl*: cryst. from dil. HCl. M.p. 184-6° decomp.

Lei, Sah, Shih, *J. Chinese Chem. Soc.*, 1935, 3, 246.

Borsche, *Ber.*, 1905, 38, 835.

**4-*p*-Tolylsemicarbazide.**

Needles from EtOH. M.p. 259-60°. Spar. sol. hot H<sub>2</sub>O.

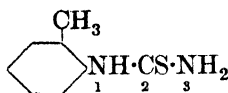
*B,HCl*: needles from dil. HCl. M.p. 242°.

Sah, Lei, *J. Chinese Chem. Soc.*, 1934, 2, 167.



**Tolyl styryl Ketone.**

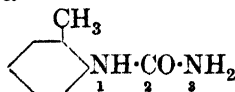
See 2-, 3-, and 4-Methylchalkone.

**o-Tolylthiourea** $C_8H_{10}N_2S$ 

MW, 166

Cryst. from  $H_2O$ . M.p.  $162^\circ$  ( $158^\circ$ ). Sol. EtOH, boiling  $H_2O$ . Spar. sol.  $Et_2O$ .1-N-Me: see *unsym.*-Methyl-o-tolylthiourea.3-N-Me: see *sym.*-Methyl-o-tolylthiourea.3-N-Et: *sym.*-ethyl-o-tolylthiourea. Prisms from EtOH. M.p.  $83-4^\circ$ .3-N-Phenyl: see *N*-Phenyl-*N'*-o-tolylthiourea.Dyson, Hunter, *J. Soc. Chem. Ind.*, 1926, **45**, 81r.Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 371.Staats, *Ber.*, 1880, **13**, 136.**m-Tolylthiourea.**Prisms from EtOH. M.p.  $110-11^\circ$ . Sol. EtOH,  $Et_2O$ . Mod. sol. hot  $H_2O$ .3-N-Phenyl: see *N*-Phenyl-*N'*-m-tolylthiourea.3-N-Propionyl: prisms from EtOH. M.p.  $86-7^\circ$ .

See first reference above and also

Heller, Bauer, *J. prakt. Chem.*, 1902, **65**, 366, 377.**p-Tolylthiourea.**Plates from EtOH. M.p.  $188^\circ$  ( $182^\circ$ ). Mod. sol. hot EtOH. Spar. sol. cold  $H_2O$ .3-N-Me: see *sym.*-Methyl-p-tolylthiourea.3-N-Et: *sym.*-ethyl-p-tolylthiourea. Prisms from EtOH. M.p.  $95-6^\circ$ .3-N-Isoamyl: *sym.*-isoamyl-p-tolylthiourea. Needles from EtOH. M.p.  $217^\circ$  decomp.3-N-Phenyl: see *N*-Phenyl-*N'*-p-tolylthiourea.I.G., F.P., 762,310, (*Chem. Zentr.*, 1934, **II**, 1992).Staats, *Ber.*, 1880, **13**, 136.Clermont, Wehrlin, *Bull. soc. chim.*, 1876, **26**, 126.**o-Tolylurea** $C_8H_{10}ON_2$ 

MW, 150

Leaflets from EtOH. M.p.  $190-1^\circ$  ( $182^\circ$ ). Sol. EtOH,  $Et_2O$ . Insol. cold  $H_2O$ .3-N-Phenyl: see *N*-Phenyl-*N'*-o-tolylurea.3-N-o-Nitrophenyl: cryst. from EtOH. M.p.  $189^\circ$ .3-N-Acetyl: needles from EtOH. M.p.  $168-9^\circ$ .3-N-Isobutyryl: needles from EtOH. M.p.  $134-5^\circ$ .3-N-Palmityl: needles from EtOH. M.p.  $98^\circ$ .3-N-Stearyl: m.p.  $94-5^\circ$ .3-N-Benzoyl: needles from AcOH. M.p.  $210^\circ$ .Lei, Sah, Shih, *J. Chinese Chem. Soc.*, 1935, **3**, 246.v. Braun, *Ber.*, 1908, **41**, 2152.Walther, Wlodkowski, *J. prakt. Chem.*, 1899, **59**, 273.**m-Tolylurea.**Leaflets from  $H_2O$ . M.p.  $142^\circ$ .3-N-Phenyl: see *N*-Phenyl-*N'*-m-tolylurea.1-N-Nitroso: yellow needles from  $Et_2O$ -pet. ether. M.p.  $80^\circ$ .Walther, Wlodkowski, *J. prakt. Chem.*, 1899, **59**, 275.Pierron, *Bull. soc. chim.*, 1906, **35**, 1200.**p-Tolylurea.**Needles from  $H_2O$ . M.p.  $182-3^\circ$  ( $172^\circ$ ). Sol. EtOH.1-N-Me: see *unsym.*-Methyl-p-tolylurea.3-N-Phenyl: see *N*-Phenyl-*N'*-p-tolylurea.3-N-Acetyl: needles from EtOH. M.p.  $199-200^\circ$ .3-N-Isobutyryl: needles from EtOH. M.p.  $138-9^\circ$ .3-N-Palmityl: m.p.  $89-90^\circ$ .3-N-Benzoyl: needles from EtOH. M.p.  $222-3^\circ$ .1-N-Nitroso: yellow cryst. from  $Et_2O$ -pet. ether. M.p.  $83^\circ$ .Walther, Wlodkowski, *J. prakt. Chem.*, 1899, **59**, 275.Sah, *Science Reports National Tsing-Hua University*, 1934, **2**, 227.Thate, *Rec. trav. chim.*, 1929, **48**, 116.**p-Tolylurethane.**See under *p*-Tolylcarbamic Acid.**p-Tolyl p-xylyl Ketone.**

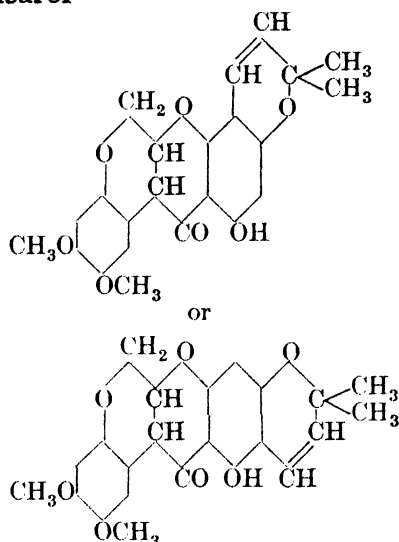
See 4:4'-Dimethyldeoxybenzoin.

**Tolysin.**

See under 6-Methyl-2-phenylquinoline-4-carboxylic Acid.

**Torulin.**See Vitamin B<sub>1</sub>.

## Toxicarol



Suggested structures

 $C_{23}H_{22}O_7$ 

MW, 410

Obtained from derris root. Greenish-yellow plates from EtOH. M.p. 218–20°. Properties closely related to those of rotenone.

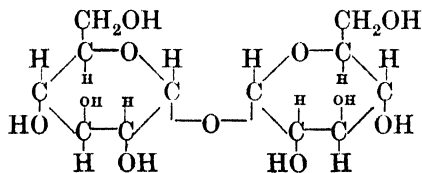
*Monoacetyl deriv.*: cryst. from  $Me_2CO$ -pet. ether. M.p. 182.5°.

*Benzoyl deriv.*: needles from  $C_6H_6-CHCl_3$ . M.p. 202°.

Heyes, Robertson, *J. Chem. Soc.*, 1935, 681.

Butenandt, Hilgetag, *Ann.*, 1933, 506, 169.

Clark, *J. Am. Chem. Soc.*, 1930, 52, 2461.

Trehalose (*Myose*) $C_{12}H_{22}O_{11}$ 

MW, 342

Occurs in fungi, moulds, ergot, algæ, yeast, etc. Prisms +  $2H_2O$  from EtOH. M.p. hydrate 97°, anhyd. 210° (203°). Sweet taste.  $[\alpha]_D^{20}$  hydrate + 178.3° in  $H_2O$ ,  $[\alpha]_D^{20}$  anhyd. + 197.0° in  $H_2O$ . Sol.  $H_2O$ . Spar. sol. EtOH. Insol.  $Et_2O$ . Does not reduce Fehling's. Non-fermentable. Does not form an osazone. Hyd. by dil. acids  $\rightarrow$  glucose.  $HNO_3 \rightarrow$  oxalic acid.

*Hexa-acetyl*: m.p. 93–6°.  $[\alpha]_D^{19} + 158.3^\circ$  in  $CHCl_3$ .

*Octa-acetyl*: cryst. from EtOH. M.p. 97–8° (97°, 96–8°, 80°, 70–5°, 100–2° after drying in vacuo).  $[\alpha]_D^{20} + 162.3^\circ$  in  $CHCl_3$ .

*Octa-nitrate*: laminæ from EtOH. M.p. 124°. Decomp. at 136°.  $[\alpha]_D^{18} + 173.8^\circ$  in AcOH.

*Octa-Me ether*: yellow liq. B.p. 170°/0.03 mm.  $[\alpha]_D^{20} + 199.8^\circ$  in  $C_6H_6$ .

Schlubach, Maurer, *Ber.*, 1925, 58, 1183.

Berthelot, *Ann. chim. phys.*, 1859, 55, 272, 291.

v. Lippmann, *Ber.*, 1912, 45, 3431.

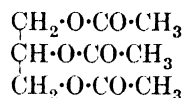
Will, Lenze, *Ber.*, 1898, 31, 85.

Pangborn, Anderson, *J. Biol. Chem.*, 1933, 101, 105.

Bredereck, *Ber.*, 1930, 63, 959.

Harding, *Sugar*, 1923, 25, 476, (*Chem. Abstracts*, 1924, 18, 78).

**Triacetin** (*Triacetyl glycerol*, *glycerol triacetate*)

 $C_9H_{14}O_6$ 

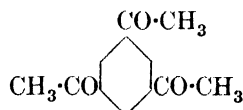
MW, 218

Colourless liq. B.p. 258–60°, 172°/40 mm. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Sol.  $H_2O$  to 70% at 15°. Prac. insol.  $CS_2$ , ligroin.  $D_4^{25}$  1.1562.

Perkin, Simonsen, *J. Chem. Soc.*, 1905, 87, 858.

Böttiger, *Ann.*, 1891, 263, 359.

**1 : 3 : 5-Triacetobenzene** (*1 : 3 : 5-Triacetylbenzene*)

 $C_{12}H_{12}O_3$ 

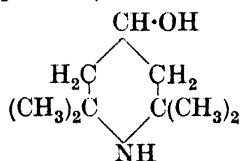
MW, 204

Needles from EtOH or AcOH. M.p. 163°. Sol. AcOH. Spar. sol.  $H_2O$ , EtOH,  $Et_2O$ .  $HNO_3 \rightarrow$  trimesic acid.

Viguier, *Compt. rend.*, 1911, 153, 1232.

Claisen, Stylos, *Ber.*, 1888, 21, 1145.

**Triacetonealkamine** (*4-Hydroxy-2 : 2 : 6 : 6-tetramethylpiperidine*)

 $C_9H_{19}ON$ 

MW, 157

Plates from  $\text{Et}_2\text{O}$ , hydrated cryst. from  $\text{H}_2\text{O}$ . M.p.  $129^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Sublimes.

*N-Me*:  $\text{C}_{10}\text{H}_{21}\text{ON}$ . MW, 171. Hydrated plates from  $\text{H}_2\text{O}$ . M.p. anhyd.  $74^\circ$ . B.p.  $125^\circ/12$  mm. Very sol. warm  $\text{H}_2\text{O}$ .

*O*: *N-Di-Me*:  $\text{C}_{11}\text{H}_{23}\text{ON}$ . MW, 185. Oil. B.p.  $217-21^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ .

*O-Benzoyl*: needles from  $\text{EtOH.Aq.}$  M.p.  $97-8^\circ$ . *B.HCl*: m.p.  $240^\circ$ .

*O*: *N-Dibenzoyl*: cryst.  $+1\text{H}_2\text{O}$  from  $\text{EtOH.Aq.}$  M.p.  $200^\circ$ .

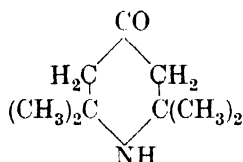
*N-Nitroso*: pale yellow needles from  $\text{C}_6\text{H}_6$ -pet. ether. M.p.  $93^\circ$ .

Orthner, *Ann.*, 1927, 456, 252.

Harries, *Ann.*, 1918, 417, 121.

Clarke, Francis, *Ber.*, 1912, 45, 2060.

**Triacetonamine** (4-*Keto*-2:2:6:6-tetramethylpiperidine, 2:2:6:6-tetramethyl- $\gamma$ -piperidone)



$\text{C}_9\text{H}_{17}\text{ON}$

MW, 155

Plates  $+1\text{H}_2\text{O}$  from  $\text{Et}_2\text{O}$ , m.p.  $58^\circ$ . Anhyd. needles from dry  $\text{Et}_2\text{O}$ , m.p.  $34.9^\circ$ . B.p.  $205^\circ$ . Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Red.  $\rightarrow$  triacetonalkamine. Forms unstable Na deriv.

$\text{B}_2\text{H}_2\text{CrO}_4$ : yellow prisms. Decomp. at  $105^\circ$ .

*Oxime*: prisms from  $\text{EtOH}$ . M.p.  $153^\circ$ .

*Semicarbazone*: cryst. from  $\text{EtOH}$ . M.p.  $219-20^\circ$ .

*N-Me*: 1:2:2:6:6-pentamethyl- $\gamma$ -piperidone.  $\text{C}_{10}\text{H}_{19}\text{ON}$ . MW, 169. Yellow liq. with unpleasant odour. B.p.  $200^\circ$  decomp.,  $122^\circ/23$  mm. *B.HI*: m.p.  $172^\circ$ .

*N-Et*:  $\text{C}_{11}\text{H}_{21}\text{ON}$ . MW, 183. Liq.  $\text{B}_2\text{H}_2\text{PtCl}_6$ : orange prisms from  $\text{H}_2\text{O}$ . M.p.  $157-8^\circ$  decomp.

*N-Allyl*:  $\text{C}_{12}\text{H}_{21}\text{ON}$ . MW, 195. Liq.  $\text{B}_2\text{H}_2\text{PtCl}_6$ : orange-yellow plates or prisms. M.p.  $148^\circ$ .

*N-Benzyl*:  $\text{C}_{16}\text{H}_{23}\text{ON}$ . MW, 245. *B.HCl*: plates or prisms from  $\text{EtOH-Et}_2\text{O}$ . M.p.  $137-8^\circ$ .  $\text{B}_2\text{H}_2\text{PtCl}_6$ : orange-red cryst. M.p.  $147-8^\circ$  decomp.

*N-Bromo*:  $\text{C}_9\text{H}_{16}\text{ONBr}$ . MW, 234. Cryst. from pet. ether. M.p.  $44^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ .

*N-Nitroso*: needles from  $\text{H}_2\text{O}$  or  $\text{EtOH.Aq.}$  M.p.  $72-3^\circ$ . Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Volatile in steam. Sublimes.

*N-OH*: triacetone-hydroxylamine.  $\text{C}_9\text{H}_{17}\text{O}_2\text{N}$ . MW, 171. Cryst. from pet. ether. M.p.  $50-1^\circ$ . Sol. dil. alkalis and acids. Reduces  $\text{NH}_3\text{AgNO}_3$ . *B.HI*: m.p.  $180^\circ$ . *Oxime*: cryst. from pet. ether. M.p.  $126-7^\circ$ .

Francis, *J. Chem. Soc.*, 1927, 2897.

Clarke, Francis, *Ber.*, 1912, 45, 2064.

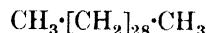
Harries, *Ann.*, 1918, 417, 171.

Harries, Lehmann, *Ber.*, 1897, 30, 232, 2736.

### Triacetone-hydroxylamine.

See under Triacetonamine.

### Triacontane



$\text{C}_{30}\text{H}_{62}$

MW, 422

Constituent of many mineral oils, of the wax coating of apple skins and of numerous flowers, *Arnica montana*, *Linaria vulgaris*, etc. Plates from  $\text{C}_6\text{H}_6$ . M.p.  $66^\circ$ . B.p.  $235^\circ/1$  mm. Very sol. hot  $\text{C}_6\text{H}_6$ . Spar. sol. hot  $\text{EtOH}$ .

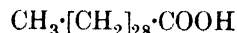
Landa, *Chem. Abstracts*, 1929, 23, 4667.

Gascard, *Ann. chim.*, 1921, 15, 332.

### Triacontane-1-carboxylic Acid.

See Melissic Acid.

### n-Triacontanic Acid



$\text{C}_{30}\text{H}_{60}\text{O}_2$

MW, 452

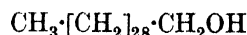
Colourless plates from  $\text{Me}_2\text{CO}$ . M.p.  $93.5-94^\circ$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ .

*Me ester*:  $\text{C}_{31}\text{H}_{62}\text{O}_2$ . MW, 466. Colourless plates from pet. ether. M.p.  $71.5^\circ$ .

*Et ester*:  $\text{C}_{32}\text{H}_{64}\text{O}_2$ . MW, 480. Colourless plates from  $\text{EtOH}$ . M.p.  $70.5^\circ$ .

Robinson, *J. Chem. Soc.*, 1934, 1544.

### n-Triacontanol (1-Hydroxytriacontane)



$\text{C}_{30}\text{H}_{62}\text{O}$

MW, 438

Plates from  $\text{C}_6\text{H}_6$ . M.p.  $86.5^\circ$ .

*Acetyl*: plates from pet. ether. M.p.  $69^\circ$ .

Robinson, *J. Chem. Soc.*, 1934, 1545.

### Triallylamine



$\text{C}_9\text{H}_{15}\text{N}$

MW, 137

Liq. with unpleasant odour. B.p.  $155-6^\circ$ .  $D^{20}_4$  0.8094.

Grosheintz, *Bull. soc. chim.*, 1879, 31, 391.

### Triallyl phosphate.

See under Phosphoric Acid.

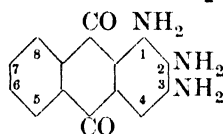
**Triallyltricyanamide** (*Sinamin*, *allylcyanamide*)

$C_{12}H_{18}N_6$  MW, 246

Cryst. +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p.  $100^\circ$  with loss of  $H_2O$ . Strongly alkaline.

Will, *Ann.*, 1844, **52**, 15.

### 1 : 2 : 3-Triaminoanthraquinone



$C_{14}H_{11}O_2N_3$  MW, 253

Black needles from  $PhNO_2$ . M.p.  $325^\circ$  decomp. Sol. conc.  $H_2SO_4 \rightarrow$  red sol.

Scholl, Eberle, Tritsch, *Monatsh.*, 1911, **32**, 1044.

Scholl, Schneider, Eberle, *Ber.*, 1904, **37**, 4438.

### 1 : 2 : 4-Triaminoanthraquinone.

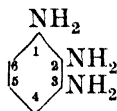
Bluish-red powder. Does not melt below  $300^\circ$ .

1 : 2 : 4 - N - Triphenyl : 1 : 2 : 4 - trianilino-anthraquinone.  $C_{32}H_{23}O_2N_3$ . MW, 481. Black needles from  $PhNO_2$  or Py. Spar. sol. EtOH. Conc.  $H_2SO_4 \rightarrow$  yellow sol.  $\rightarrow$  red on warming.

Terres, *Monatsh.*, 1921, **41**, 608.

Bayer, D.R.P., 151,511, (*Chem. Zentr.*, 1904, I, 1507).

### 1 : 2 : 3-Triaminobenzene



$C_6H_9N_3$  MW, 123

Cryst. M.p. up to  $103^\circ$ . B.p.  $336^\circ$ . Sol.  $H_2O$ , EtOH,  $Et_2O$ . Reacts strongly alkaline.  $FeCl_3 \rightarrow$  violet  $\rightarrow$  brown ppt. Sol. in conc.  $H_2SO_4$  + trace  $HNO_3 \rightarrow$  dark blue col. Reduces  $NH_3 \cdot AgNO_3$ . Boiling AcOH  $\rightarrow$  4-acetyl-amino-2-methylbenzimidazole.

2-N-Phenyl : see 2 : 6-Diaminodiphenylamine.

Salkowski, *Ann.*, 1872, **163**, 23.

### 1 : 2 : 4-Triaminobenzene.

Plates from  $CHCl_3$ . M.p. below  $100^\circ$ . B.p. about  $340^\circ$ . Sol.  $H_2O$ , EtOH. Mod. sol.  $CHCl_3$ . Spar. sol.  $Et_2O$ . Turns brown in air.  $FeCl_3 \rightarrow$  red col.

1-N-Di-Me : 2 : 4-diamino-N-dimethylaniline.  $C_8H_{13}N_3$ . MW, 151. Needles from ligroin. M.p.  $44^\circ$ . B.p.  $218-19^\circ/90$  mm.,  $178^\circ/22$  mm. Sol.  $H_2O$ . Turns dark blue in air.

Aq. sol. + ox. agents  $\rightarrow$  red sol.  $B, 2HCl$  : cryst. M.p.  $225^\circ$ .  $B, 2HBr$  : m.p.  $207^\circ$  decomp.  $B, 2HI$  : plates +  $\frac{1}{2}EtOH$  from EtOH. M.p.  $190^\circ$  decomp. 2 : 4-N-Diacetyl : cryst. +  $\frac{1}{2}H_2O$  from  $H_2O$ , m.p.  $82^\circ$ ; cryst. from  $C_6H_6$  or  $AcOEt$ , m.p.  $153^\circ$ .

2 : 4-N-Tetra-Me :  $C_{10}H_{17}N_3$ . MW, 179. Liq. B.p.  $209.4^\circ/112$  mm.,  $180.5^\circ/45$  mm.  $D_4^{20}$  1.0203. Has caustic action.  $B, 2HCl$  : cryst. powder. M.p.  $164^\circ$ .  $B, 2HBr$  : m.p.  $179^\circ$ .  $B, 2HI$  : prisms. M.p.  $175^\circ$  decomp. Picrate : m.p.  $169^\circ$  decomp. 1-N-Acetyl : plates from ligroin. M.p.  $85^\circ$ . 1-N-Benzenesulphonyl : prisms from ligroin. M.p.  $84^\circ$ .

1 : 2 : 4-N-Hexa-Me :  $C_{12}H_{21}N_3$ . MW, 207. Liq. B.p.  $210^\circ/136$  mm.,  $184^\circ/40$  mm. Trimethiodide : needles +  $2MeOH$  from MeOH. M.p.  $164.5^\circ$  decomp.

1-N-Phenyl : see 2 : 4-Diaminodiphenylamine.

2 : 4-N-Diphenyl : 4-amino-3-anilinodiphenylamine.  $C_{18}H_{17}N_3$ . MW, 275. Needles from  $C_6H_6$ -ligroin. M.p.  $107^\circ$ . Spar. sol.  $H_2O$ , ligroin. Sol.  $Et_2O$ ,  $C_6H_6$ .

1-N-Acetyl : 2 : 4-diaminoacetanilide. Prisms. M.p.  $158-9^\circ$ .

1 : 4-N-Diacetyl : cryst. M.p.  $231-2^\circ$ .

1 : 2 : 4-N-Tribenzoyl : needles from AcOH. M.p.  $260^\circ$ .

Hinsberg, *Ber.*, 1886, **19**, 1253.

Wurster, Sendtner, *Ber.*, 1879, **12**, 1806.

### 1 : 3 : 5-Triaminobenzene.

Free base not isolated.

1 : 3 : 5-N-Triphenyl : 1 : 3 : 5-trianilino-benzene.  $C_{24}H_{21}N_3$ . MW, 351. Needles from EtOH. M.p.  $193^\circ$ . Sol.  $Et_2O$ . Spar. sol. cold EtOH,  $C_6H_6$ . Conc.  $H_2SO_4 \rightarrow$  violet-red col. on warming.  $B, HCl$  : yellow powder. M.p. below  $100^\circ$ .  $B, H_2PtCl_6$  : yellow ppt. M.p.  $251^\circ$  decomp. 1 : 3 : 5-N-Trinitroso : brown needles from EtOH. M.p.  $264-5^\circ$ . 1 : 3 : 5-N-Triacetyl : needles from EtOH. M.p.  $172-3^\circ$ .

1 : 3 : 5-N-Tri-p-tolyl :  $C_{27}H_{27}N_3$ . MW, 393. Needles from EtOH. M.p.  $186-7^\circ$ . Spar. sol. cold EtOH. Conc.  $H_2SO_4 \rightarrow$  bluish-green on warming. 1 : 3 : 5-N-Trinitroso : needles from EtOH. M.p.  $233-4^\circ$ . 1 : 3 : 5-N-Triacetyl : needles from EtOH. M.p.  $192-3^\circ$ . 1 : 3 : 5-N-Tribenzoyl : prisms from EtOH. M.p.  $281-2^\circ$ .

1 : 3 : 5-N-Triacetyl : plates from EtOH. M.p.  $208^\circ$ .

1 : 3 : 5-N-Tribenzoyl : needles from EtOH- $C_6H_6$ . Does not melt below  $350^\circ$ .

Minunni, *Gazz. chim. ital.*, 1890, **20**, 322.

Hepp, *Ann.*, 1882, **215**, 348.

## 2 : 3 : 5-Triaminobenzoic Acid

 $C_7H_9O_2N_3$ 

MW, 167

Cryst. from  $H_2O$ . Chars on heating evolving  $NH_3$ . Very sol. hot  $H_2O$ . Spar. sol. hot EtOH. Insol. Et<sub>2</sub>O. Aq. sol. rapidly becomes red.  $FeCl_3 \rightarrow$  brown ppt.

2-N-Phenyl : 3 : 5-diamino-2-anilinobenzoic acid, 4 : 6-diaminodiphenylamine-2-carboxylic acid.  $C_{13}H_{13}O_2N_3$ . MW, 243. Cryst. M.p. 237–8° decomp. Very sol. EtOH, AcOH. Spar. sol. Et<sub>2</sub>O,  $C_6H_6$ , hot  $H_2O$ .

Cohn, Schifferes, *Chem. Zentr.*, 1902, I, 1293.

Griess, *Ber.*, 1882, 15, 2199.

## 2 : 4 : 6-Triaminobenzoic Acid.

$HNO_3 \rightarrow$  intense yellow sol., violet on neutralisation. Hydrochloride + boiling  $H_2O \rightarrow$  phloroglucinol.

Hydrochloride : prisms from  $H_2O$ .

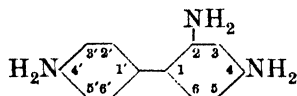
Cassella, D.R.P., 102,358, (*Chem. Zentr.*, 1899, I, 1263).

## 3 : 4 : 5-Triaminobenzoic Acid.

Needles +  $\frac{1}{2}H_2O$  from hot  $H_2O$ . Loses  $H_2O$  at 100°. Sol. hot  $H_2O$ . Spar. sol. cold  $H_2O$ . Insol. hot EtOH. Reacts acid. Dist.  $\rightarrow$  1 : 2 : 3-triaminobenzene. Sol. in conc.  $H_2SO_4$  + trace  $HNO_3 \rightarrow$  bluish-green  $\rightarrow$  dark blue col.

Salkowski, *Ann.*, 1872, 163, 12.

## 2 : 4 : 4' - Triaminodiphenyl (2-Amino-benzidine)

 $C_{12}H_{13}N_3$ 

MW, 199

Needles. M.p. 134°.

4 : 4'-N-Di-p-toluenesulphonyl : m.p. 198°.

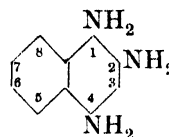
Tauber, *Ber.*, 1890, 23, 798.

## 2 : 5 : 4'-Triaminodiphenyl.

5-N-Di-Me :  $C_{14}H_{17}N_3$ . MW, 227. Cryst. M.p. 87–9°. Sol. EtOH, dil. min. acids. Spar. sol.  $H_2O$ , ligroin.  $FeCl_3 \rightarrow$  violet  $\rightarrow$  blue col. Picrate : yellow prisms. M.p. 127°. 2 : 4'-N-Diacetyl : needles from EtOH. Aq. M.p. 233°.

Jacobson, Kunz, *Ann.*, 1898, 303, 354.

## 1 : 2 : 4-Triaminonaphthalene

 $C_{10}H_{11}N_3$ 

MW, 173

1-N-Phenyl : 2 : 4-diamino-1-anilinonaphthalene, 2 : 4-diamino-1-N-phenylnaphthylamine.  $C_{16}H_{15}N_3$ . MW, 249. Needles from  $C_6H_6$ . M.p. 190°. Sol. AcOH,  $C_6H_6$ . Very spar. sol. ligroin, Et<sub>2</sub>O.

1 : 2 : 4-N-Triphenyl : 1 : 2 : 4-trianilino-naphthalene.  $C_{28}H_{23}N_3$ . MW, 401. Needles from EtOH or  $C_6H_6$ -ligroin. M.p. 148°.

1-N-p-Tolyl : 2 : 4-diamino-1-N-p-tolyl-naphthylamine.  $C_{17}H_{17}N_3$ . MW, 263. Needles from  $C_6H_6$ . M.p. 176–7°. Very readily oxidised. Resinifies in air.

1 : 2 : 4-N-Tri-p-tolyl :  $C_{31}H_{29}N_3$ . MW, 443. Needles from EtOH. M.p. 159–60°.

1-N-Acetyl : 2 : 4-diamino-1-acetnaphthalide. Brown needles. M.p. 189°.

1 : 2 : 4-N-Triacetyl : white cryst. M.p. 301°.

1-N-Benzenesulphonyl : needles from toluene-pet. ether. M.p. 195–7°.

Panizzon-Favre, *Gazz. chim. ital.*, 1924, 54, 826.

Ullmann, Bruck, *Ber.*, 1908, 41, 3937.

Fischer, Hepp, *Ann.*, 1890, 256, 250.

## 1 : 2 : 5-Triaminonaphthalene.

B<sub>2</sub>HCl : cryst. Turns brown in  $H_2O$ .

1 : 2 : 5-N-Tribenzoyl : yellow powder. M.p. 268°.

Finzi, *Chem. Abstracts*, 1925, 19, 2661.

## 1 : 2 : 6-Triaminonaphthalene.

2-N-Me :  $C_{11}H_{13}N_3$ . MW, 187. 2-N-p-Toluenesulphonyl : prisms from  $C_6H_6$ -pet. ether. Decomp. at 185°.

1 : 2 : 6-N-Triacetyl : needles from AcOH. M.p. 280° decomp.

1 : 2 : 6-N-Tribenzoyl : needles from AcOH. M.p. 277°.

2-N-p-Toluenesulphonyl : needles from toluene. M.p. 190°.

Loewe, *Ber.*, 1890, 23, 2544.

## 1 : 3 : 6-Triaminonaphthalene.

Sol.  $H_2O$ . Acid. sol. + nitrite  $\rightarrow$  deep brown col.

Sulphate : needles. Spar. sol.  $H_2O$ .

Kalle, D.R.P., 89,061.

**1 : 3 : 7-Triaminonaphthalene.**

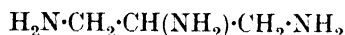
Sol. H<sub>2</sub>O, EtOH. Aq. sol. + HNO<sub>3</sub> → deep brown col. Salt sols + FeCl<sub>3</sub> → violet col.  
*Sulphate*: cryst. Spar. sol. hot H<sub>2</sub>O.

Kalle, D.R.P., 90,905.

**1 : 3 : 8-Triaminonaphthalene.**

*B,3HI*: needles.

Aguiar, Lautemann, *Bull. soc. chim.*, 1865, 3, 263.

**1 : 2 : 3-Triaminopropane**

C<sub>3</sub>H<sub>11</sub>N<sub>3</sub> MW, 89

Viscous oil. B.p. 190°, 92–3°/9 mm. Sol. H<sub>2</sub>O.

*B,3HCl, H<sub>2</sub>O*: plates. M.p. 250°.

*B,3HCl, AuCl<sub>3</sub>*: yellow prisms. M.p. 210–12°.

*B,3HCl, PtCl<sub>4</sub>*: yellow needles. M.p. 220° decomp.

*B,2HCl, HI*: cryst. from H<sub>2</sub>O. M.p. 303–4° decomp.

*B,3HBr*: m.p. 307–10°.

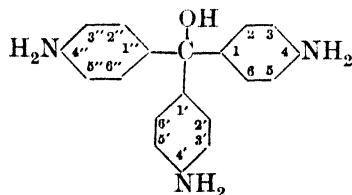
*Triacetyl*: cryst. from EtOH. M.p. 200–2°.

*Tribenzoyl*: m.p. 217–18°.

*Picrate*: yellow needles. Does not melt below 270°.

Pope, Mann, *Compt. rend.*, 1924, 178, 2085.

Brackebusch, *Ber.*, 1873, 6, 1290.

**4 : 4' : 4''-Triaminotriphenylcarbinol**  
(*Pararosanine* base)

C<sub>19</sub>H<sub>19</sub>ON<sub>3</sub> MW, 305

Colourless plates. M.p. about 205°. Turns red in air. Very spar. sol. H<sub>2</sub>O. Sol. EtOH. Insol. Et<sub>2</sub>O. Heat of comb. C<sub>v</sub> 2481.0 Cal., C<sub>p</sub> 2483.5 Cal. Red. → *paraleucaniline*. H<sub>2</sub>O at 270° → 4 : 4'-dihydroxybenzophenone. KCN → triaminotriphenylacetone nitrile.

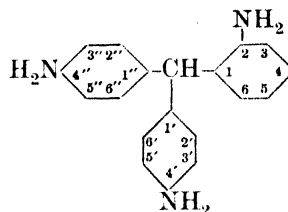
*Me ether*: C<sub>20</sub>H<sub>21</sub>ON<sub>3</sub>. MW, 319. Plates + 1Et<sub>2</sub>O from Et<sub>2</sub>O. m.p. 105°; plates + 1C<sub>6</sub>H<sub>6</sub> from C<sub>6</sub>H<sub>6</sub>, m.p. 135°.

4 : 4' : 4''-N-*Triacetyl*: needles from Me<sub>2</sub>CO-Et<sub>2</sub>O. M.p. 192°.

Wieland, Scheuing, *Ber.*, 1921, 54, 2527. M.L.B., D.R.P. 300,467, (*Chem. Zentr.*, 1917, II, 579).

Zimmermann, Müller, *Ber.*, 1885, 18, 997. Fischer, *Ber.*, 1882, 15, 678; 1880, 13, 2205.

Fischer, *Ann.*, 1878, 194, 274.

**2 : 4' : 4''-Triaminotriphenylmethane**

C<sub>19</sub>H<sub>19</sub>N<sub>3</sub> MW, 289

Cryst. from EtOH. M.p. 165°.

Renouf, *Ber.*, 1883, 16, 1305.

**3 : 4' : 4''-Triaminotriphenylmethane.**

Needles + 1C<sub>6</sub>H<sub>6</sub> from C<sub>6</sub>H<sub>6</sub>, m.p. 145°; cryst. from Et<sub>2</sub>O-petrol, m.p. 150°. Sol. EtOH.

Fischer, Ziegler, *Ber.*, 1880, 13, 672.

**4 : 4' : 4''-Triaminotriphenylmethane**  
(*Paraleucaniline*).

Plates from H<sub>2</sub>O, EtOH or C<sub>6</sub>H<sub>6</sub>. M.p. 202.5° (208°). Ox. → *pararosanine* base. Di-azotise + EtOH → triphenylmethane.

4 : 4' : 4''-N-*Triacetyl*: reddish plates from EtOH. M.p. 201°.

*B, C<sub>6</sub>H<sub>3</sub>(NO<sub>2</sub>)<sub>3</sub>-1 : 3 : 5*: black prisms. M.p. 140°.

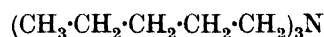
Fischer *et al.*, *J. prakt. Chem.*, 1909, 79, 563; *Ann.*, 1878, 194, 268.

**Triaminotriazine.**

See Melamine.

**3 : 4 : 5-Triamino-1 : 2 : 4-triazole.**

See Guanazine.

**Tri-*n*-amylamine**

C<sub>15</sub>H<sub>33</sub>N MW, 227

Liq. B.p. 240–5°, 130°/14 mm.

Marvel, Scott, Amstutz, *J. Am. Chem. Soc.*, 1929, 51, 3640.

Mailhe, *Compt. rend.*, 1918, 166, 997.

**Tri-active-amine**

$\text{C}_{15}\text{H}_{33}\text{N}$   $(\text{CH}_3\cdot\text{CH}_2\cdot\overset{\text{CH}_3}{\underset{|}{\text{CH}}}\cdot\text{CH}_2)_3\text{N}$  MW, 227  
 Liq. B.p. 230–7°.  $D_{20}^{25}$  0.7964.

Plimpton, *J. Chem. Soc.*, 1881, **39**, 335.

**Triamyl phosphate.**

See under Phosphoric Acid.

**Trianilinoanthraquinone.**

See under Triaminoanthraquinone.

**Trianilinobenzene.**

See under Triaminobenzene.

**Trianilinomethane.**

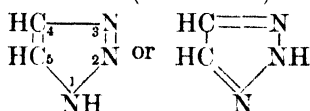
See under Orthoformic Acid.

**Trianilinonaphthalene.**

See under Triaminonaphthalene.

**Triazo-**

See Azido-, and individual azides.

**1 : 2 : 3-Triazole (Osoatriazole)**

$\text{C}_2\text{H}_3\text{N}_3$  MW, 69

M.p. 23°. B.p. 203°/739 mm. Sol. ord. org. solvents. Insol. ligroin. Hygroscopic.

1-N-Phenyl : see 1-Phenyl-1 : 2 : 3-triazole.

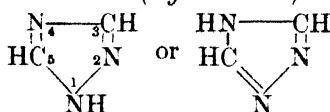
1-N-Benzyl :  $\text{C}_9\text{H}_9\text{N}_3$ . MW, 159. Cryst. M.p. 61°. B.p. 180–3°/16 mm.

1-N-Benzoyl : needles from  $\text{CHCl}_3$  or  $\text{Et}_2\text{O}$ . M.p. 100–2°.

Pechmann, *Bayer, Ber.*, 1909, **42**, 673.

Zincke, *Ann.*, 1900, **311**, 317.

Dimroth, *Ber.*, 1902, **35**, 1044.

**1 : 2 : 4-Triazole (Pyrrodiazole)**

$\text{C}_2\text{H}_3\text{N}_3$  MW, 69

Needles from  $\text{Et}_2\text{O}$  or  $\text{C}_6\text{H}_6$ . M.p. 120–1°. B.p. 260°. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Forms comps. with metals and metallic salts.

$B, \text{HCl}$  : plates. M.p. 169°.

Oxalate : cryst. from  $\text{H}_2\text{O}$ . Decomp. at 251°.

$B_2, \text{H}_2\text{PtCl}_6$  : needles. M.p. 73–5°.

1-Phenyl : see 1-Phenyl-1 : 2 : 4-triazole.

3-Phenyl : see 3-Phenyl-1 : 2 : 4-triazole.

Pellizzari, *Gazz. chim. ital.*, 1911, **41**, 20 ;

*Atti accad. Lincei*, 1902, **11**, 20.

Paolini, *Baj, Gazz. chim. ital.*, 1931, **61**, 557.

Bladin, *Ber.*, 1892, **25**, 745.

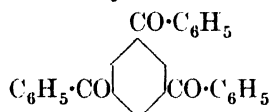
**Tribenzamide**

$\text{C}_{21}\text{H}_{15}\text{O}_3\text{N}$   $\text{C}_6\text{H}_5\cdot\text{CO}\cdot\text{N}(\text{CO}\cdot\text{C}_6\text{H}_5)_2$  MW, 329

Needles from  $\text{EtOH}$ . M.p. 207–8° (202°). Sublimes. Heat. of comb. 2425.5 Cal. Hyd. by  $\text{KOH}$ .

Titherley, *J. Chem. Soc.*, 1904, **85**, 1187.

Wheeler, Walden, Metcalf, *J. Am. Chem. Soc.*, 1898, **20**, 73.

**1 : 3 : 5-Tribenzoylbenzene**

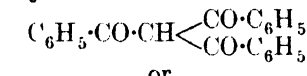
$\text{C}_{27}\text{H}_{18}\text{O}_3$  MW, 390

Needles from  $\text{EtOH}$ . M.p. 118–19°.

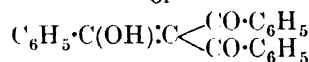
Trioxime : m.p. 198°.

Monophenylhydrazone : m.p. 84°.

Claisen, *Ann.*, 1894, **281**, 307.

**Tribenzoylmethane**

or



$\text{C}_{22}\text{H}_{16}\text{O}_3$  MW, 328

$\alpha$ - or Enol form :

Cryst. M.p. (in Jena glass) 155°, (in soft glass) 240–5°. More sol. than keto-form.  $\text{FeCl}_3 \longrightarrow$  red col.  $\text{EtOH}$  or  $\text{CH}_3\text{COCl} \longrightarrow$  keto-form.

$\beta$ - or Keto form :

Needles from  $\text{EtOH}$  or  $\text{Me}_2\text{CO}$ . M.p. 223–6° (231°), in Jena glass 245–50°.  $\text{FeCl}_3 \longrightarrow$  brownish-red col. Conc.  $\text{H}_2\text{SO}_4 \longrightarrow$  yellow col.  $\text{NaOEt} \longrightarrow$  enol-form.

Dieckmann, *Ber.*, 1916, **49**, 2209.

Abell, *J. Chem. Soc.*, 1912, **101**, 998.

Claisen, *Ann.*, 1896, **291**, 92.

**Tribenzylamine**

$\text{C}_{21}\text{H}_{21}\text{N}$   $(\text{C}_6\text{H}_5\cdot\text{CH}_2)_3\text{N}$  MW, 287

Plates or prisms from  $\text{Et}_2\text{O}$ . M.p. 92°. B.p. 230°/13 mm. Sol. hot  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ . Hot  $\text{HCl} \longrightarrow$  dibenzylamine + benzyl chloride.

$B, \text{HCl}$  : prisms from  $\text{EtOH}$ . M.p. 227–8°.

$B, \text{HBr}$  : prisms. M.p. 208°.

$B, \text{HI}$  : prisms from  $\text{AcOH}$ . M.p. 185–8° (178°).

$B, \text{HNO}_3$  : cryst. from  $\text{EtOH}$ . M.p. 120°.

$B, \text{H}_2\text{SO}_4$  : prisms from  $\text{EtOH}$ . M.p. 106–7°.

*B. Benzenesulphonic acid* : needles from  $\text{H}_2\text{O}$ . M.p.  $200^\circ$ .

*B. p-Toluenesulphonic acid* : m.p.  $205.2-207.7^\circ$ .

*Methochloride* : needles from  $\text{H}_2\text{O}$ , plates from EtOH. M.p.  $202^\circ$ .

*Methiodide* : needles from  $\text{H}_2\text{O}$ , plates from EtOH. M.p.  $184^\circ$ .

*Ethiodide* : needles from  $\text{H}_2\text{O}$ . M.p.  $190^\circ$ .

Scheibler, Beiser, Cobler, Schmidt, *Ber.*, 1934, **67**, 1509.

Mason, *J. Chem. Soc.*, 1893, **63**, 1314.

**Tribenzyl phosphate.**

See under Phosphoric Acid.

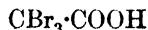
**Tribromoacetaldehyde.**

See Bromal.

**Tribromoacetanilide.**

See under Tribromoaniline.

**Tribromoacetic Acid**



$\text{C}_2\text{H}_2\text{O}_2\text{Br}_3$  MW, 297

White cryst. M.p.  $131^\circ$ . B.p.  $245^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Boiling  $\text{H}_2\text{O}$  or EtOH  $\rightarrow$  bromoform.

*Et ester* :  $\text{C}_4\text{H}_5\text{O}_2\text{Br}_3$ . MW, 325. B.p.  $225^\circ$ ,  $148^\circ/73$  mm.  $D_{20}^{20}$  2.230.  $n_D^{19}$  1.54377.

*Bromide* :  $\text{C}_2\text{OBr}_4$ . MW, 360. B.p.  $210-15^\circ$ ,  $88-90^\circ/12$  mm.

*Amide* :  $\text{C}_2\text{H}_5\text{ONBr}_3$ . MW, 296. M.p.  $121-2^\circ$ . Sol.  $\text{Et}_2\text{O}$ , hot EtOH. Sublimes.

*Nitrile* :  $\text{C}_2\text{NBr}_3$ . MW, 278. Oil. B.p.  $170^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

Schäffer, *Ber.*, 1871, **4**, 370.

**1 : 1 : 1-Tribromoacetone**

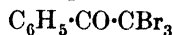


$\text{C}_3\text{H}_3\text{OBr}_3$  MW, 295

B.p.  $255^\circ$  decomp.  $\text{NH}_3 \rightarrow$  bromoform.

Étard, *Compt. rend.*, 1892, **114**, 754.

**$\omega$ -Tribromoacetophenone**

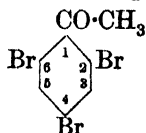


$\text{C}_8\text{H}_5\text{OBr}_3$  MW, 357

B.p.  $174^\circ/14$  mm.

Myddleton, Barrett, Seager, *J. Am. Chem. Soc.*, 1930, **52**, 4409.

**2 : 4 : 6-Tribromoacetophenone**



$\text{C}_8\text{H}_5\text{OBr}_3$  MW, 357

Needles from EtOH.Aq. M.p.  $93.5^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .

Fuchs, *Monatsh.*, 1915, **36**, 136.

**3 : 4 : 5-Tribromoacetophenone.**

Cryst. from EtOH. M.p.  $134-5^\circ$ . Sol. EtOH,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{Et}_2\text{O}$ , pet. ether, hot  $\text{H}_2\text{O}$ .

*Phenylhydrazone* : yellow cryst. from pet. ether. M.p.  $129-34^\circ$  decomp.

*Semicarbazone* : cryst. from AcOH. M.p.  $265^\circ$  decomp.

*Azine* : m.p.  $300^\circ$ .

Bruining, *Rec. trav. chim.*, 1922, **41**, 655.

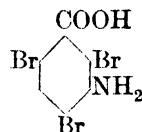
**Tribromoacet-toluidide.**

See under Tribromotoluidine.

**Tribromo-*o*-aminobenzoic Acid.**

See Tribromoanthranilic Acid.

**2 : 4 : 6-Tribromo-*m*-aminobenzoic Acid**



$\text{C}_7\text{H}_4\text{O}_2\text{NBr}_3$  MW, 374

Needles from  $\text{H}_2\text{O}$ . M.p.  $170.5^\circ$ . Sol. EtOH. Spar. sol.  $\text{H}_2\text{O}$ . Decomp. on dist.  $\rightarrow$  2 : 4 : 6-tribromoaniline.

*Me ester* :  $\text{C}_8\text{H}_6\text{O}_2\text{NBr}_3$ . MW, 388. M.p.  $96-7^\circ$ .

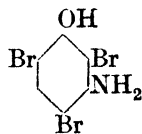
*Et ester* :  $\text{C}_9\text{H}_8\text{O}_2\text{NBr}_3$ . MW, 402. M.p.  $61-2^\circ$ .

*Nitrile* :  $\text{C}_7\text{H}_3\text{N}_2\text{Br}_3$ . MW, 355. Needles from EtOH. M.p.  $177-8^\circ$ . Sol. EtOH,  $\text{CHCl}_3$ ,  $\text{Me}_2\text{CO}$ .

Sudborough, Karvé, *Chem. Abstracts*, 1920, **14**, 3652.

Sudborough, Lloyd, *J. Chem. Soc.*, 1899, **75**, 589.

**2 : 4 : 6-Tribromo-*m*-aminophenol**



$\text{C}_6\text{H}_4\text{ONBr}_3$  MW, 346

Needles from pet. ether. M.p.  $119^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. pet. ether.  $\text{FeCl}_3 \rightarrow$  green col.

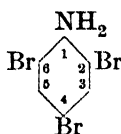
O : N : N-Triacetyl : m.p.  $136^\circ$ .

*p-Toluenesulphonate* : prisms from EtOH. M.p.  $146-7^\circ$ .

Bamberger, *Ber.*, 1915, **48**, 1355.



## 2 : 4 : 6-Tribromoaniline

 $C_6H_4NBr_3$ 

MW, 330

Needles from  $C_6H_6$  or EtOH. M.p.  $122^\circ$  ( $120^\circ$ ). Sol.  $Et_2O$ , hot EtOH. Insol.  $H_2O$ .  $Sn + HCl \rightarrow$  2 : 4-dibromoaniline. Hot conc.  $HCl \rightarrow$  2 : 4 : 6-trichloroaniline.

*B, HBr*: needles. M.p.  $195-6^\circ$ .

*N-Me*: 2 : 4 : 6-tribromomethylaniline.

$C_7H_6NBr_3$ . MW, 344. Needles from MeOH.

M.p.  $39^\circ$ . *N-Acetyl*: m.p.  $101^\circ$ .

*N-Di-Me*: 2 : 4 : 6-tribromodimethylaniline.

$C_8H_8NBr_3$ . MW, 358. Oil. B.p.  $301^\circ/750$  mm.

*N-Et*: 2 : 4 : 6-tribromoethylaniline.  $C_8H_8NBr_3$ . MW, 358. Needles from AcOH. M.p.  $45^\circ$ .

*N-Formyl*: 2 : 4 : 6-tribromoformanilide. Needles from EtOH. M.p.  $221.5^\circ$ .

*N-Acetyl*: 2 : 4 : 6-tribromoacetanilide. Needles from EtOH. M.p.  $232^\circ$ .

*N-Diacetyl*: needles from EtOH. M.p.  $127-8^\circ$ .

*N-Propionyl*: prisms from EtOH. M.p.  $203^\circ$ .

*N-Benzoyl*: 2 : 4 : 6-tribromobenzanilide. Needles from EtOH. M.p.  $204^\circ$ .

Silberstein, *J. prakt. Chem.*, 1883, 27, 101.

Asinger, *J. prakt. Chem.*, 1935, 142, 299.

## 3 : 4 : 5-Tribromoaniline.

Cryst. from EtOH. M.p.  $123^\circ$ .

*N-Acetyl*: 3 : 4 : 5-tribromoacetanilide.

Needles from  $Et_2O$ . M.p.  $255-6^\circ$ .

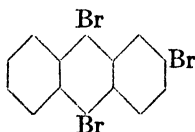
*N-Benzoyl*: 3 : 4 : 5-tribromobenzanilide. Cryst. from EtOH. M.p.  $210^\circ$ .

Asinger, *J. prakt. Chem.*, 1935, 142, 300.

## Tribromoanisole.

See under Tribromophenol.

## 2 : 9 : 10-Tribromoanthracene

 $C_{14}H_7Br_3$ 

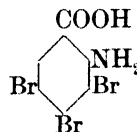
MW, 415

Cryst. from amyl alcohol. M.p.  $171^\circ$ . Very sol.  $C_6H_6$ . Spar. sol. EtOH.  $CrO_3 \rightarrow$  2-bromoanthraquinone.

Barnett, Cook, *J. Chem. Soc.*, 1925, 1490.

Grandmougin, *Compt. rend.*, 1921, 173, 1176.

## 3 : 4 : 5-Tribromoanthranilic Acid (3 : 4 : 5-Tribromo-o-aminobenzoic acid)

 $C_7H_4O_2NBr_3$ 

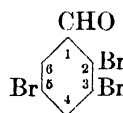
MW, 374

Cryst. from EtOH.Aq. Decomp. at  $240^\circ$ .

I.G., D.R.P., 528,115, (*Chem. Zentr.*, 1931, II, 1927).

See also Lesser, Weiss, *Ber.*, 1913, 46, 3941.

## 2 : 3 : 5-Tribromobenzaldehyde

 $C_7H_3OBr_3$ 

MW, 343

Cryst. from pet. ether. M.p.  $114^\circ$ . Sol. EtOH. Insol.  $H_2O$ .

Blanksma, *Chem. Zentr.*, 1912, II, 1964.

## 2 : 4 : 6-Tribromobenzaldehyde.

Cryst. from EtOH. M.p.  $99^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Oxime*: needles from EtOH.Aq. M.p.  $175^\circ$ .

See previous reference.

## 3 : 4 : 5-Tribromobenzaldehyde.

Cryst. from EtOH.Aq. M.p.  $109^\circ$ . Sol. EtOH,  $C_6H_6$ . Insol.  $H_2O$ .

*Oxime*: needles from EtOH.Aq. M.p.  $172^\circ$ .

*Phenylhydrazones*: yellow plates. M.p.  $158^\circ$ .

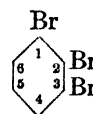
*Semicarbazones*: needles from Py. M.p.  $314^\circ$ .

*Diacetate*: cryst. from EtOH. M.p.  $100^\circ$ .

*Azine*: yellow needles +  $2H_2O$ . M.p.  $315^\circ$ .

Janse, *Rec. trav. chim.*, 1921, 40, 285.

## 1 : 2 : 3-Tribromobenzene

 $C_6H_3Br_3$ 

MW, 315

Plates from EtOH. M.p.  $87-8^\circ$ .

Körner, *Gazz. chim. ital.*, 1874, 4, 408.

Jackson, Gallivan, *Am. Chem. J.*, 1898, 20, 179.

## 1 : 2 : 4-Tribromobenzene.

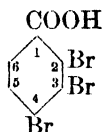
Needles with aromatic odour from EtOH. M.p.  $44-5^\circ$ . B.p.  $275^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ .

Jackson, Gallivan, *Am. Chem. J.*, 1896, 18, 241.

**1 : 3 : 5-Tribromobenzene.**

Needles or prisms from EtOH. M.p. 120°. B.p. 271°. Spar. sol. hot EtOH. Insol. H<sub>2</sub>O. NaOMe at 130° → 3 : 5-dibromophenol.

Coleman, Talbot, *Organic Syntheses*, 1933, XIII, 96.

**2 : 3 : 4-Tribromobenzoic Acid**

C<sub>7</sub>H<sub>3</sub>O<sub>2</sub>Br<sub>3</sub> MW, 359

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 197–8°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 511.

**2 : 3 : 5-Tribromobenzoic Acid.**

Needles from EtOH. M.p. 193–4°. Sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, hot EtOH. Spar. sol. C<sub>6</sub>H<sub>6</sub>. pet. ether.

*Ba salt* : cryst. + 5H<sub>2</sub>O. Very sol. H<sub>2</sub>O.

*Me ester* : C<sub>8</sub>H<sub>5</sub>O<sub>2</sub>Br<sub>3</sub>. MW, 373. Needles from EtOH.Aq. M.p. 77°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 512.

Blanksma, *Chem. Zentr.*, 1912, II, 1965.

**2 : 4 : 5-Tribromobenzoic Acid.**

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 195–6°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 515.

**2 : 4 : 6-Tribromobenzoic Acid.**

Prisms from H<sub>2</sub>O. M.p. 194°.

*Me ester* : needles from EtOH.Aq. M.p. 69°.

*Et ester* : C<sub>9</sub>H<sub>7</sub>O<sub>2</sub>Br<sub>3</sub>. MW, 387. M.p. 80°.

*Chloride* : C<sub>7</sub>H<sub>2</sub>OBr<sub>3</sub>Cl. MW, 377.5. Plates from pet. ether. M.p. 48°.

*Amide* : C<sub>7</sub>H<sub>4</sub>ONBr<sub>3</sub>. MW, 358. Prisms. M.p. 195°.

*Dimethylamide* : C<sub>9</sub>H<sub>8</sub>ONBr<sub>3</sub>. MW, 386. Prisms from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 85–6°.

*Nitrile* : C<sub>7</sub>H<sub>2</sub>NBr<sub>3</sub>. MW, 340. Needles from EtOH. M.p. 128°.

*Anilide* : needles from EtOH. M.p. 237°.

Asinger, *J. prakt. Chem.*, 1935, 142, 296.

Buning, *Rec. trav. chim.*, 1921, 40, 327.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 516.

**3 : 4 : 5-Tribromobenzoic Acid.**

Needles from C<sub>6</sub>H<sub>6</sub> or EtOH.Aq. M.p. 240°.

*Me ester* : needles from EtOH. M.p. 154°.

*Et ester* : needles. M.p. 126°.

*Chloride* : needles from pet. ether. M.p. 83°.

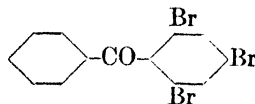
Dict. of Org. Comp.—III.

*Amide* : needles from EtOH.Aq. M.p. 200°.

*Anilide* : needles from EtOH. M.p. 220°.

Asinger, *J. prakt. Chem.*, 1935, 142, 298.

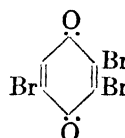
See also last reference above.

**2 : 4 : 6-Tribromobenzophenone**

C<sub>13</sub>H<sub>7</sub>OBr<sub>3</sub> MW, 419

Cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 147°.

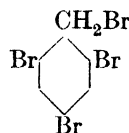
Montagne, *Rec. trav. chim.*, 1908, 27, 353.

**2 : 3 : 5-Tribromo-*p*-benzoquinone (Tri-bromoquinone)**

C<sub>6</sub>H<sub>2</sub>O<sub>2</sub>Br<sub>3</sub> MW, 345

Yellow plates from EtOH. M.p. 147°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Sublimes. NaOH → green col.

Sarauw, *Ann.*, 1881, 209, 120.

**2 : 4 : 6-Tribromobenzyl bromide (α:2:4:6-Tetrabromotoluene)**

C<sub>7</sub>H<sub>4</sub>Br<sub>4</sub> MW, 408

Needles from EtOH or AcOH. M.p. 75°. B.p. 202°/18 mm.

Asinger, *J. prakt. Chem.*, 1935, 142, 296.

**Tribromobenzyl cyanide.**

See under Tribromophenylacetic Acid.

**1 : 1 : 2-Tribromobutane**

C<sub>4</sub>H<sub>7</sub>Br<sub>3</sub> MW, 295

Yellow oil with camphor-like odour. B.p. 216.2°, 98°/14 mm. D<sub>4</sub><sup>15</sup> 2.1913. Darkens on heating at atm. press.

Kaufmann, Schweitzer, *Ber.*, 1922, 55, 264.

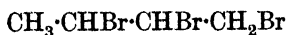
**1 : 2 : 2-Tribromobutane**

C<sub>4</sub>H<sub>7</sub>Br<sub>3</sub> MW, 295

B.p. 213.8°, 112–15°/40 mm., 90.1°/14 mm. D<sub>4</sub><sup>15</sup> 2.1761.

Lépingle, *Bull. soc. chim.*, 1926, 39, 741.

## 1 : 2 : 3-Tribromobutane



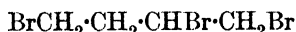
$\text{C}_4\text{H}_7\text{Br}_3$  MW, 295

B.p. 110–13°/19 mm., 100–1°/14 mm., 94–6°/6 mm.  $D_4^{16}$  2.190.  $n_D^{15}$  1.5691.

Slobodin, *Chem. Abstracts*, 1935, 29, 4732.

Delaby, *Compt. rend.*, 1923, 176, 589.

## 1 : 2 : 4-Tribromobutane



$\text{C}_4\text{H}_7\text{Br}_3$  MW, 295

B.p. 115–17°/10 mm.  $D_4^{16}$  2.234.  $n_D^{15}$  1.574.

Pariselle, *Ann. chim. phys.*, 1911, 24, 323.

## 2 : 2 : 3-Tribromobutane

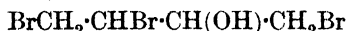


$\text{C}_4\text{H}_7\text{Br}_3$  MW, 295

B.p. 206.5°, 83–4°/11.5 mm.  $D_4^{15}$  2.1806.

Lépingle, *Bull. soc. chim.*, 1926, 39, 741.

**1 : 3 : 4-Tribromo-sec.-*n*-butyl Alcohol**  
(Erythritol tribromohydrin, 1 : 3 : 4-tribromo-2-hydroxybutane)

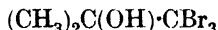


$\text{C}_4\text{H}_7\text{OBr}_3$  MW, 311

B.p. 148–50°/14 mm.

Pariselle, *Ann. chim. phys.*, 1911, 24, 405.

**1 : 1 : 1-Tribromo-*tert*.-butyl Alcohol**  
(Brometone)



$\text{C}_4\text{H}_7\text{OBr}_3$  MW, 311

Cryst. with camphor-like odour from EtOH.Aq. M.p. 167–76°.

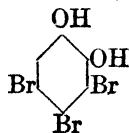
Acetyl : cryst. from EtOH. M.p. 43–4°.

Propionyl : cryst. from EtOH. M.p. 27°.

Butyryl : b.p. 144–5°/13 mm.

Aldrich, *J. Am. Chem. Soc.*, 1911, 33, 387.

## 3 : 4 : 5-Tribromocatechol



$\text{C}_6\text{H}_3\text{O}_2\text{Br}_3$  MW, 347

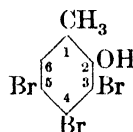
Cryst. +  $1\text{H}_2\text{O}$ . M.p. 144°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{CHCl}_3$ , pet. ether. Insol.  $\text{H}_2\text{O}$ .

*Di-Me ether* : 3 : 4 : 5-tribromoveratrol.  $\text{C}_8\text{H}_7\text{O}_3\text{Br}_3$ . MW, 375. Needles. M.p. 86–7°. Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold EtOH.

*Diacetyl* : cryst. M.p. 120°.

Frejka, Šefránek, *Chem. Zentr.*, 1936, I, 2338.

Chem. Fabrik, von Heyden, D.R.P., 207,544, (*Chem. Zentr.*, 1909, I, 1283); 215,337, (*Chem. Zentr.*, 1909, II, 1710).

3 : 4 : 5-Tribromo-*o*-cresol

$\text{C}_7\text{H}_5\text{OBr}_3$  MW, 345

Needles from petrol. M.p. 89°. Sol. EtOH,  $\text{Et}_2\text{O}$ , AcOH,  $\text{C}_6\text{H}_6$ . Spar. sol. petrol.

*Acetyl* : plates from AcOH. M.p. 106–7°.

Kohn, Aron, *Monatsh.*, 1929, 53, 49.

Janney, *Ann.*, 1913, 398, 367.

3 : 5 : 6-Tribromo-*o*-cresol.

Needles from petrol. M.p. 91°. Sol.  $\text{Et}_2\text{O}$ , AcOH,  $\text{C}_6\text{H}_6$ . Spar. sol. petrol.  $\text{NaNO}_2$  in AcOH  $\rightarrow$  3 : 4-dibromo-6-nitro-*o*-cresol.

*Me ether* :  $\text{C}_8\text{H}_7\text{OBr}_3$ . MW, 359. Needles from EtOH. M.p. 71°. B.p. 308–11°/745 mm.

*Acetyl* : cryst. from AcOH. M.p. 76–7°.

*Benzoyl* : prisms from  $\text{C}_6\text{H}_6$ . M.p. 133°.

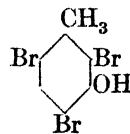
Kohn, Aron, *Monatsh.*, 1929, 53, 54.

4 : 5 : 6-Tribromo-*o*-cresol.

Needles from pet. ether. M.p. 106°.

*Me ether* : cryst. from EtOH. M.p. 105°. B.p. 320°.

Kohn Soltész, *Monatsh.*, 1925, 46, 250.

2 : 4 : 6-Tribromo-*m*-cresol

$\text{C}_7\text{H}_5\text{OBr}_3$  MW, 345

Needles from EtOH or pet. ether. M.p. 81.5–82°. Cryst. +  $1\text{AcOH}$  from AcOH. Ox.  $\rightarrow$  3 : 5-dibromotoluquinone.

*Et ether* :  $\text{C}_9\text{H}_9\text{OBr}_3$ . MW, 373. Needles from EtOH. M.p. 36°.

*Acetyl* : needles from EtOH. M.p. 68°.

*Benzoyl* : needles from EtOH. M.p. 84–5°.

*Benzenesulphonyl* : plates from EtOH. M.p. 117–117.5°.

*p*-Toluenesulphonyl : plates from EtOH. M.p. 113–14°.

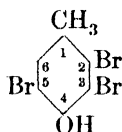
Huston, Peterson, *J. Am. Chem. Soc.*, 1933, **55**, 3882.

Jost, Richter, *Ber.*, 1923, **56**, 122.

van Erp, *Rec. trav. chim.*, 1911, **30**, 302.

Claus, Hirsch, *J. prakt. Chem.*, 1889, **39**, 59.

### 2 : 3 : 5-Tribromo-*p*-cresol



$C_7H_5OBr_3$

MW, 345

Needles from petrol. M.p. 102°. Sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ , hot  $Na_2CO_3$ -Aq. Spar. sol. petrol.

*Acetyl* : needles from ligroin. M.p. 77°.

Zincke, Wiederhold, *Ann.*, 1902, **320**, 205.

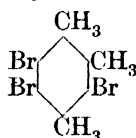
### 2 : 3 : 6-Tribromo-*p*-cresol.

*Benzoyl* : plates from  $Me_2CO$ . M.p. 120°.

*p*-Nitrobenzoyl : plates from  $Me_2CO-CHCl_3$ . Softens at 156°, m.p. 159–60°.

Jadhav, Rangwala, *Chem. Zentr.*, 1935, **I**, 2976.

3 : 5 : 6-Tribromo- $\psi$ -cumene (3 : 5 : 6-Tribromo-1 : 2 : 4-trimethylbenzene)



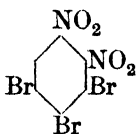
$C_9H_9Br_3$

MW, 357

Cryst. M.p. 229–30°. Sol. boiling toluene, hot AcOH. Spar. sol. hot EtOH.

Fittig, Laubinger, *Ann.*, 1869, **151**, 267.

### 3 : 4 : 5-Tribromo-*o*-dinitrobenzene



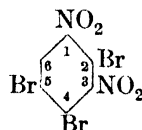
$C_6HO_4N_2Br_3$

MW, 405

Plates or prisms. M.p. 162–4°. Sol. AcOH,  $CHCl_3$ ,  $C_6H_6$ , EtOH,  $Et_2O$ , hot ligroin. Insol.  $H_2O$ . Boiling dil. NaOH  $\rightarrow$  4 : 5 : 6-tribromo-*o*-nitrophenol.

Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 586.

### 2 : 4 : 5-Tribromo-*m*-dinitrobenzene



$C_6HO_4N_2Br_3$

MW, 405

Yellow scales. M.p. 135.5°. Sol. hot EtOH,  $Et_2O$ ,  $CS_2$ . Spar. sol. cold EtOH. Red.  $\rightarrow$  5-bromo-*m*-phenylenediamine.

Jackson, Gallivan, *Ber.*, 1895, **28**, 190.

### 2 : 4 : 6-Tribromo-*m*-dinitrobenzene.

Colourless prisms or needles from EtOH. M.p. 192°. Very sol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ . Sol. hot EtOH. Spar. sol. cold EtOH. Insol.  $H_2O$ . Red.  $\rightarrow$  2 : 4 : 6-tribromo-*m*-phenylenediamine.  $Zn + HCl \rightarrow m$ -phenylenediamine.

Jackson, Koch, *Am. Chem. J.*, 1899, **21**, 519.

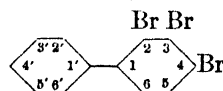
### 4 : 5 : 6-Tribromo-*m*-dinitrobenzene.

Cryst. from EtOH. M.p. 150–1°. Sol.  $Me_2CO$ , AcOH,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH, ligroin. Alc.  $NH_3$  at 100°  $\rightarrow$  5 : 6-dibromo-2 : 4-dinitroaniline.

van de Bunt, *Rec. trav. chim.*, 1929, **48**, 128.

Jackson, Earle, *Am. Chem. J.*, 1901, **26**, 51.

### 2 : 3 : 4-Tribromodiphenyl



$C_{12}H_7Br_3$

MW, 391

Needles. M.p. 225–7°.

Bellavita, *Chem. Zentr.*, 1936, **I**, 2342.

### 2 : 4 : 5-Tribromodiphenyl.

Prisms. M.p. 68°.

Bellavita, *Chem. Zentr.*, 1936, **I**, 2341.

### 2 : 4 : 6-Tribromodiphenyl.

Needles from MeOH. M.p. 64°.

Blakey, Scarborough, *J. Chem. Soc.*, 1927, 3008.

### 2 : 5 : 4'-Tribromodiphenyl.

Yellow needles from EtOH. M.p. 76°.

Bellavita, *Gazz. chim. ital.*, 1935, **65**, 643.

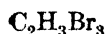
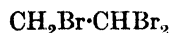
### 2 : 3' : 4'-Tribromodiphenyl.

Needles from EtOH. M.p. 91°.

Bellavita, *Gazz. chim. ital.*, 1935, **65**, 644.

**4 : 5 : 4'-Tribromodiphenyl.**

Prisms. M.p. 102°.

Bellavita, *Chem. Zentr.*, 1936, I, 2342.**1 : 1 : 2-Tribromoethane**

MW, 267

M.p. -26°. B.p. 187-8°/721 mm., 83°/18 mm.  $D_4^{20}$  2.57896.  $n_D^{20}$  1.58902. Alc. KOH  $\rightarrow$  1 : 1-dibromoethylene + bromoacetylene + acetylene.

Wurtz, *Ann.*, 1857, 104, 243.**2 : 2 : 2-Tribromoethyl Alcohol (Tribromoethanol, Avertin)**

MW, 283

Needles or prisms. M.p. 80°. B.p. 92-3°/10 mm. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, warm pet. ether. Spar. sol. H<sub>2</sub>O. Used in anaesthesia.

Chloroformyl: b.p. 103°/10 mm.

Urethane: prisms from EtOH. M.p. 86-7°. Very sol. H<sub>2</sub>O. Sol. EtOH, Et<sub>2</sub>O.

I.G., D.R.P., 565,157, (*Chem. Zentr.*, 1933, I, 1514); E.P., 370,490, (*Chem. Zentr.*, 1932, II, 3303).

Willstätter, Duisberg, *Ber.*, 1923, 56, 2284.

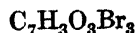
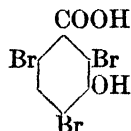
**Tribromoethylene**

MW, 265

B.p. 162.5°, 75°/15 mm.  $D_4^{20}$  2.708.  $n_D^{25}$  1.62475. O  $\rightarrow$  dibromoacetyl bromide. In air  $\rightarrow$  pentabromoethane. Zn + EtOH  $\rightarrow$  acetylene + bromoacetylene.

Hg comp.: prisms from CHCl<sub>3</sub>. M.p. 141°.Dehn, *J. Am. Chem. Soc.*, 1912, 34, 286.**Tribromohydrin.**

See 1 : 2 : 3-Tribromopropane.

**2 : 4 : 6-Tribromo-*m*-hydroxybenzoic Acid**

MW, 375

Needles +  $\frac{1}{2}$  H<sub>2</sub>O from H<sub>2</sub>O. Anhyd. at 100°. M.p. 146-7°. Sol. EtOH, Et<sub>2</sub>O, AcOH. Mod. sol. CHCl<sub>3</sub>. Insol. C<sub>6</sub>H<sub>6</sub>. Boiling HI  $\rightarrow$  *m*-hydroxybenzoic acid.

Me ether: 2 : 4 : 6-tribromo-*m*-methoxybenzoic

acid. C<sub>8</sub>H<sub>5</sub>O<sub>3</sub>Br<sub>3</sub>. MW, 389. Cryst. from AcOH.Aq. M.p. 154°.

Me ester: C<sub>8</sub>H<sub>5</sub>O<sub>3</sub>Br<sub>3</sub>. MW, 389. Plates and needles from ligroin. M.p. 119-21°. Sol. most org. solvents.

Amide: C<sub>7</sub>H<sub>4</sub>O<sub>2</sub>NBr<sub>3</sub>. MW, 374. M.p. 221°.

Nitrile: C<sub>7</sub>H<sub>2</sub>ONBr<sub>3</sub>. MW, 356. Yellow needles from ligroin. M.p. 168°. Spar. sol. ligroin. Acetyl: yellowish-white plates from EtOH.Aq. M.p. 156-8°.

Acetyl: cryst. from MeOH or CHCl<sub>3</sub>-pet. ether. M.p. 192-3°.

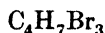
Bull, Ross, Fuson, *J. Am. Chem. Soc.*, 1935, 57, 764.

Luck, Hosaeus, *Monatsh.*, 1933, 62, 186.

Leulier, Pinet, *Bull. soc. chim.*, 1927, 41, 1362.

Werner, *Bull. soc. chim.*, 1886, 46, 276.**Tribromohydroxybenzyl bromide.**

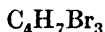
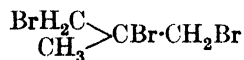
See Tetrabromocresol.

**1 : 1 : 2-Tribromoisobutane**

MW, 295

B.p. 205-6° decomp., 121-4°/57 mm., 110-14°/15 mm.  $D_4^{21}$  2.0169.

Norton, Williams, *Am. Chem. J.*, 1887, 9, 89.

**1 : 2 : 3-Tribromoisobutane**

MW, 295

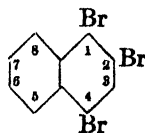
B.p. 173-83°/235 mm., 108-9°/18 mm.  $D_4^{24}$  2.2106.  $n_D^{14}$  1.57012.

Mereshkowski, *Chem. Zentr.*, 1914, I, 2160.

Norton, Williams, *Am. Chem. J.*, 1887, 9, 88.

**Tribromomethane.**

See Bromoform.

**1 : 2 : 4-Tribromonaphthalene**

MW, 365

Needles. M.p. 113-14°. Very sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, Me<sub>2</sub>CO. Ox.  $\rightarrow$  phthalic acid.

Prager, *Ber.*, 1885, 18, 2164.

**1 : 2 : 6-Tribromonaphthalene.**

Needles from EtOH. M.p. 118°. Very sol. EtOH, Et<sub>2</sub>O, ligroin. Slowly volatile in steam. Sublimes.

Claus, Philipson, *J. prakt. Chem.*, 1891, **43**, 53.

**1 : 3 : 6-Tribromonaphthalene.**

Needles from EtOH, plates from H<sub>2</sub>O. M.p. 98° (86.5°). Sol. most. org. solvents. Sublimes.

Franzen, Stäuble, *J. prakt. Chem.*, 1920, **101**, 58.

Claus, Jäck, *J. prakt. Chem.*, 1898, **57**, 17.

**1 : 4 : 5-Tribromonaphthalene.**

Needles. M.p. 86°. Very sol. EtOH.

Salkind, Belikoff, *Ber.*, 1931, **64**, 958.

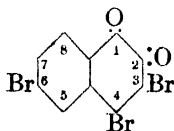
John, *Bull. soc. chim.*, 1877, **28**, 515.

**1 : 4 : 6-Tribromonaphthalene.**

Needles from EtOH. M.p. 86-7°. Very sol. EtOH, Et<sub>2</sub>O.

Salkind, Belikoff, *Ber.*, 1931, **64**, 959.

Glaser, *Ann.*, 1865, **135**, 43.

**3 : 4 : 6-Tribromo-1 : 2-naphthoquinone**

C<sub>10</sub>H<sub>3</sub>O<sub>2</sub>Br<sub>3</sub>

MW, 395

Red needles from C<sub>6</sub>H<sub>6</sub>. M.p. 193°.

Quinoxaline with *o*-phenylenediamine: yellow needles from AcOH. M.p. 250°.

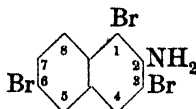
Fries, Schimmelschmidt, *Ann.*, 1930, **484**, 274.

**3 : 5 : 6-Tribromo-1 : 2-naphthoquinone.**

Red needles from C<sub>6</sub>H<sub>6</sub>-petrol. M.p. 184°. Mod. sol. EtOH, AcOH, petrol.

Quinoxaline with *o*-phenylenediamine: yellow needles from toluene. M.p. 271°.

Fries, Schimmelschmidt, *Ann.*, 1930, **484**, 282.

**1 : 3 : 6-Tribromo-2-naphthylamine**

C<sub>10</sub>H<sub>6</sub>NBr<sub>3</sub>

MW, 380

Pale red cryst. from EtOH. M.p. 143°. Very sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. EtOH, pet. ether. Ox. → 4-bromophthalic acid.

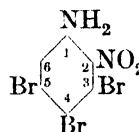
*N*-Acetyl: 1 : 3 : 6-tribromo-2-acetnaphthalide. Needles from EtOH. M.p. 250-1°.

*N*-Diacetyl: plates from EtOH. M.p. 159°.

*N*-*p*-Toluenesulphonyl: needles from AcOH. M.p. 184°.

Bell, *J. Chem. Soc.*, 1932, 2734.

Franzen, Stäuble, *J. prakt. Chem.*, 1920, **101**, 61.

**3 : 4 : 5-Tribromo-*o*-nitroaniline**

C<sub>6</sub>H<sub>3</sub>O<sub>2</sub>N<sub>2</sub>Br<sub>3</sub>

MW, 375

Yellow needles. M.p. 134°. Sol. EtOH. Spar. sol. hot H<sub>2</sub>O.

*N*-Acetyl: 3 : 4 : 5-tribromo-2-nitroacetanilide. Needles from EtOH or C<sub>6</sub>H<sub>6</sub>, prisms from AcOEt. M.p. 229-30°.

Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 581.

**4 : 5 : 6-Tribromo-*o*-nitroaniline.**

Yellow needles. M.p. 166°. Very sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Sol. MeOH, Et<sub>2</sub>O, AcOH. Spar. sol. EtOH. Insol. H<sub>2</sub>O, ligroin.

*N*-Me: C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>N<sub>2</sub>Br<sub>3</sub>. MW, 389. M.p. 128°.

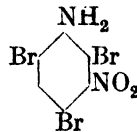
*N*-Et: C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>N<sub>2</sub>Br<sub>3</sub>. MW, 403. M.p. 130°.

*N*-Phenyl: 4 : 5 : 6-tribromo-2-nitrodiphenylamine. C<sub>12</sub>H<sub>7</sub>O<sub>2</sub>N<sub>2</sub>Br<sub>3</sub>. MW, 451. Light red prisms or yellow needles. M.p. 138-9°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH, AcOH. Insol. H<sub>2</sub>O, ligroin.

*N*-Acetyl: needles. M.p. 221°.

Jackson, Fiske, *Am. Chem. J.*, 1903, **30**, 74.

Blanksma, *Rec. trav. chim.*, 1902, **21**, 414.

**2 : 4 : 6-Tribromo-*m*-nitroaniline**

C<sub>6</sub>H<sub>3</sub>O<sub>2</sub>N<sub>2</sub>Br<sub>3</sub>

MW, 375

Yellow needles from EtOH. M.p. 120-5°. Very sol. EtOH.

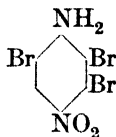
*N*-Acetyl: 2 : 4 : 6-tribromo-3-nitroacetanilide. Cryst. from EtOH or AcOH, needles from C<sub>6</sub>H<sub>6</sub>. M.p. 208-9° (216-17°).

*N*-Diacetyl: cryst. from EtOH. M.p. 168-9° (175-6°).

N-*Chloro* :  $C_6H_4O_3N_2ClBr_3$ . MW, 409.5.  
Prisms from  $CHCl_3$ -pet. ether. M.p. 159°.

Jackson, Jones, *Am. Chem. J.*, 1913, **49**, 48.

### 2 : 3 : 6-Tribromo-*p*-nitroaniline



$C_6H_3O_2N_2Br_3$  MW, 375

Yellow needles. M.p. 159°. Spar. sol. cold EtOH. Insol.  $H_2O$ . Slowly volatile in steam.

Orton, *J. Chem. Soc.*, 1902, **81**, 499.

### Tribromonitroanisole.

See under Tribromonitrophenol.

### 2 : 3 : 4-Tribromonitrobenzene



$C_6H_3O_2NBr_3$  MW, 360

Cryst. from EtOH. M.p. 85.4°. Very sol.  $Et_2O$ , AcOEt,  $C_6H_6$ . Mod. sol. EtOH. Slowly volatile in steam. Sublimes with difficulty.

Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 583.

### 2 : 3 : 5-Tribromonitrobenzene.

Needles. M.p. 81° (119.5°). Volatile in steam. Red.  $\rightarrow$  2 : 3 : 5-tribromoaniline.

Claus, Wallbaum, *J. prakt. Chem.*, 1897, **56**, 58.

### 2 : 3 : 6-Tribromonitrobenzene.

Colourless plates or prisms from EtOH- $Et_2O$ . Sublimes at 185° without melting. Very spar. sol. EtOH.

Körner, *Jahresber. Fortschr. Chem.*, 1875, 314.

### 2 : 4 : 5-Tribromonitrobenzene.

Needles from EtOH. M.p. 93.5°. Sol.  $Et_2O$ , hot EtOH,  $CS_2$ . Dist. without decomp. Volatile in steam. Sublimes. Red.  $\rightarrow$  2 : 4 : 5-tribromoaniline.

Körner, *Jahresber. Fortschr. Chem.*, 1875, 313.

### 2 : 4 : 6-Tribromonitrobenzene.

Colourless prisms from  $CHCl_3$ . M.p. 124.5°. B.p. about 177°/11 mm. Sol.  $Et_2O$ , boiling

AcOH,  $CHCl_3$ . Spar. sol. boiling EtOH. Red.  $\rightarrow$  2 : 4 : 6-tribromoaniline.

Orton, *J. Chem. Soc.*, 1903, **83**, 806.

Hantzsch, Blagden, *Ber.*, 1900, **33**, 2553.

### 3 : 4 : 5-Tribromonitrobenzene.

Colourless cryst. from EtOH- $Et_2O$ . M.p. 112°. Volatile in steam. Sublimes. Red.  $\rightarrow$  3 : 4 : 5-tribromoaniline. NaOH  $\rightarrow$  2 : 6-dibromo-*p*-nitrophenol.

Asinger, *J. prakt. Chem.*, 1935, **142**, 299.

Körner, Contardi, *Atti accad. Lincei*, 1906, **15**, 585.

### Tribromonitrodiphenylamine.

See under Tribromonitroaniline.

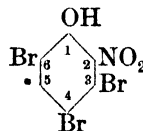
### Tribromonitromethane.

See Bromopierin.

### Tribromonitrophenetole.

See under Tribromonitrophenol.

### 3 : 4 : 6-Tribromo-*o*-nitrophenol



$C_6H_3O_3NBr_3$  MW, 376

Pale yellow plates from H-COOH.Aq. M.p. 127°.

*Me ether* : 3 : 4 : 6-tribromo-*o*-nitroanisole.  $C_7H_4O_3NBr_3$ . MW, 390. Colourless prisms from H-COOH.Aq. M.p. 72°.

Hodgson, Walker, Smith, *J. Chem. Soc.*, 1933, 1055.

### 4 : 5 : 6-Tribromo-*o*-nitrophenol.

Yellow prisms from  $C_6H_6$  or MeOH. M.p. 123°. Very sol.  $Me_2CO$ , hot  $C_6H_6$ ,  $CHCl_3$ . Sol. EtOH,  $Et_2O$ , AcOH. Insol. cold  $C_6H_6$ .

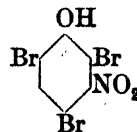
*Me ether* : 4 : 5 : 6-tribromo-*o*-nitroanisole. Needles from EtOH or H-COOH. M.p. 109°. Very sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH, AcOH, ligroin. Insol.  $H_2O$ .

*Et ether* : 4 : 5 : 6-tribromo-*o*-nitrophenetole.  $C_8H_5O_3NBr_3$ . MW, 404. Cryst. M.p. 74°. Rapidly turns brown in air.

See previous reference and also

Jackson, Fiske, *Am. Chem. J.*, 1903, **30**, 71.

### 2 : 4 : 6-Tribromo-*m*-nitrophenol



$C_6H_3O_3NBr_3$

MW, 376

Cryst. from ligroin. M.p. 89–90°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. warm H<sub>2</sub>O. Decomposes cold aq. carbonates. Forms spar. sol. Ag and Ba salts. Red. → *m*-aminophenol.

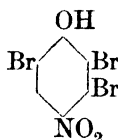
*Me ether*: 2 : 4 : 6-tribromo-*m*-nitroanisole. C<sub>7</sub>H<sub>4</sub>O<sub>3</sub>NBr<sub>3</sub>. MW, 390. Plates from EtOH. M.p. 82°.

*Et ether*: 2 : 4 : 6-tribromo-*m*-nitrophenetole. C<sub>8</sub>H<sub>6</sub>O<sub>3</sub>NBr<sub>3</sub>. MW, 404. Prisms from Et<sub>2</sub>O. M.p. 79°. Very sol. Et<sub>2</sub>O.

*Propionyl*: scales from EtOH.Aq. M.p. 70–1°.

*p*-Toluenesulphonyl: needles from EtOH. M.p. 146–7°.

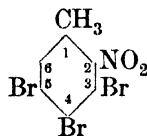
Henley, Turner, *J. Chem. Soc.*, 1930, 935.  
Bamberger, *Ber.*, 1915, 48, 1344.

2 : 3 : 6-Tribromo-*p*-nitrophenol

C<sub>6</sub>H<sub>2</sub>O<sub>3</sub>NBr<sub>3</sub> MW, 376

Pale yellow needles from hot H<sub>2</sub>O. M.p. 151° decomp.

Hodgson, Smith, *J. Chem. Soc.*, 1932, 505.

3 : 4 : 5-Tribromo-*o*-nitrotoluene

C<sub>7</sub>H<sub>4</sub>O<sub>2</sub>NBr<sub>3</sub> MW, 374

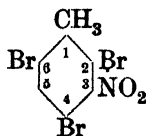
Cryst. from EtOH. M.p. 104–5°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 515.

3 : 5 : 6-Tribromo-*o*-nitrotoluene.

Cryst. from EtOH. M.p. 88.5–90° (93°). Sol. hot EtOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 512.

2 : 4 : 6-Tribromo-*m*-nitrotoluene

C<sub>7</sub>H<sub>4</sub>O<sub>2</sub>NBr<sub>3</sub> MW, 374

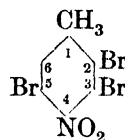
Cryst. M.p. 74–75.5°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 515.

2 : 5 : 6-Tribromo-*m*-nitrotoluene.

Yellow needles from EtOH. M.p. 91–2°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 514.

2 : 3 : 5-Tribromo-*p*-nitrotoluene

C<sub>7</sub>H<sub>4</sub>O<sub>2</sub>NBr<sub>3</sub> MW, 374

Cryst. from EtOH. M.p. 67–8°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 512.

2 : 3 : 6-Tribromo-*p*-nitrotoluene.

Cryst. from AcOH. M.p. 106.5–107°.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 514.

## Tribromophenetole.

See under Tribromophenol.

## 2 : 3 : 4-Tribromophenol



C<sub>6</sub>H<sub>3</sub>OBr<sub>3</sub> MW, 331

Prisms from H·COOH.Aq. M.p. 95°.

*Me ether*: 2 : 3 : 4-tribromoanisole. C<sub>7</sub>H<sub>5</sub>OBr<sub>3</sub>. MW, 345. M.p. 106°.

Hodgson, Walker, Nixon, *J. Chem. Soc.*, 1933, 1054.

## 2 : 3 : 5-Tribromophenol.

Needles or plates from H<sub>2</sub>O or ligroin. M.p. 94–5°. Very sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, hot ligroin, alkalis. Spar. sol. H<sub>2</sub>O. Volatile in steam. FeCl<sub>3</sub> → brownish-violet col.

*Me ether*: 2 : 3 : 5-tribromoanisole. M.p. 82°. B.p. 305–12°.

Bamberger, Kraus, *Ber.*, 1906, 39, 4251.

Kohn, Karlin, *Monatsh.*, 1927, 48, 599.

## 2 : 4 : 5-Tribromophenol.

Needles from dichloroethane–pet. ether. M.p. 87°.

*Me ether*: 2 : 4 : 5-tribromoanisole. Needles. M.p. 105°. B.p. 306–9°/775 mm.

*Benzoyl*: m.p. 99°.

*o*-Bromo-*p*-toluenesulphonyl: plates from EtOH. M.p. 107–8°.

Hodgson, Walker, Nixon, *J. Chem. Soc.*, 1933, 1054.

Turner, Henley, *J. Chem. Soc.*, 1930, 933.



**2 : 4 : 6-Tribromophenol (Bromol).**

Needles from EtOH, prisms from  $C_6H_6$ , cryst. + 1AcOH from AcOH. M.p.  $94^\circ$ . Sol. 14,000 parts  $H_2O$  at  $15^\circ$ . Very sol. EtOH. Sublimes.  $NaNO_2$  in AcOH  $\rightarrow$  4 : 6-dibromo-*o*-nitrophenol. Forms spar. sol.  $NH_4$  salt. Stronger antiseptic than phenol or thymol.

*Me ether*: 2 : 4 : 6-tribromoanisole.  $C_7H_5OBr_3$ . MW, 345. Needles from EtOH. M.p.  $88^\circ$ . Sol. 100 parts  $H_2O$  at  $15^\circ$ .

*Et ether*: 2 : 4 : 6-tribromophenetole.  $C_8H_7OBr_3$ . MW, 359. Prisms from EtOH. M.p.  $72-3^\circ$ .

*Propyl ether*:  $C_9H_9OBr_3$ . MW, 373. Needles from EtOH. M.p.  $33-4^\circ$ .

*Allyl ether*:  $C_9H_7OBr_3$ . MW, 371. Needles. M.p.  $77^\circ$ .

*p*-Nitrobenzyl ether:  $C_{13}H_9O_3NBr_3$ . MW, 466. M.p.  $163.5^\circ$ .

*Acetyl*: colourless plates or needles from EtOH. M.p.  $87^\circ$ .

*Propionyl*: needles. M.p.  $65^\circ$ .

*Benzoyl*: m.p.  $81^\circ$ .

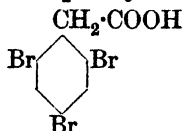
*p*-Bromobenzenesulphonyl: cryst. from EtOH. M.p.  $139-40^\circ$ .

*p*-Toluenesulphonyl: cryst. from EtOH. M.p.  $113^\circ$ .

van Erp, *Rec. trav. chim.*, 1911, **30**, 280.

Orton, Coates, Burdett, *J. Chem. Soc.*, 1907, **91**, 47.

Körner, *Ann.*, 1866, **137**, 203.

**2 : 4 : 6-Tribromophenylacetic Acid**

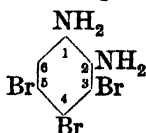
$C_8H_5O_2Br_3$  MW, 373

M.p.  $157-8^\circ$ .

*Amide*:  $C_8H_6ONBr_3$ . MW, 372. M.p.  $162-3^\circ$ .

*Nitrile*: 2 : 4 : 6-tribromobenzyl cyanide.  $C_8H_4NBr_3$ . MW, 354. M.p.  $138-9^\circ$ . Sol. EtOH. Insol.  $H_2O$ .

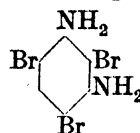
Henraut, *Chem. Zentr.*, 1924, II, 1342.

**3 : 4 : 5-Tribromo-*o*-phenylenediamine**

$C_6H_5N_2Br_3$  MW, 345

Cryst. from EtOH. M.p.  $91^\circ$ . Very sol.  $Et_2O$ ,  $Me_2CO$ . Sol. EtOH, AcOH,  $C_6H_6$ . Insol.  $H_2O$ , ligroin. Turns brown in air.

Jackson, *Am. Chem. J.* 1903, **30**, 78.

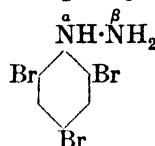
**2 : 4 : 6-Tribromo-*m*-phenylenediamine**

$C_6H_5N_2Br_3$  MW, 345

Needles from EtOH. M.p.  $158^\circ$ . Very sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. ligroin.  $Zn + HCl \rightarrow m$ -phenylenediamine.

1 : 3-N-Diacetyl: plates from AcOH. Does not melt below  $330^\circ$ .

Jackson, Calvert, *Am. Chem. J.*, 1896, **18**, 470.

**2 : 4 : 6-Tribromophenylhydrazine**

$C_6H_5N_2Br_3$  MW, 345

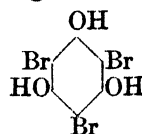
Needles from pet. ether. M.p.  $146^\circ$ . Sol. boiling AcOH. Spar. sol. boiling EtOH.

$\beta$ -N-Acetyl: prisms from MeOH. M.p.  $188^\circ$ .

$\alpha$  :  $\beta$ -N-Diacetyl: needles from EtOH. M.p.  $144-5^\circ$ .

$\beta$ -N-Benzoyl: yellow needles from EtOH. M.p.  $172^\circ$ .

Chattaway, Vonderwahl, *J. Chem. Soc.*, 1915, **107**, 1507.

**Tribromophloroglucinol**

$C_6H_3O_3Br_3$  MW, 363

Cryst. +  $3H_2O$  from  $H_2O$ . M.p. anhyd.  $152-3^\circ$  ( $149-50^\circ$ ).  $Sn + HCl \rightarrow$  phloroglucinol.

*Me ether*:  $C_7H_5O_3Br_3$ . MW, 377. Needles from  $C_6H_6$ . M.p.  $123^\circ$ . Very sol.  $H_2O$ , EtOH,  $CHCl_3$ . *Diacetyl*: needles from EtOH. M.p.  $112-14^\circ$ .

*Tri-Me ether*:  $C_9H_9O_3Br_3$ . MW, 405. Needles from EtOH. M.p.  $145^\circ$ .

*Di-Et ether*:  $C_{10}H_{11}O_3Br_3$ . MW, 419. Needles from AcOH. Aq. M.p.  $63-5^\circ$ .

*Tri-Et ether*:  $C_{12}H_{15}O_3Br_3$ . MW, 447. Needles from AcOH. M.p.  $102-4^\circ$ .

*Monoacetyl*: needles from  $C_6H_6$ -pet. ether. M.p.  $169^\circ$ .

*Triacetyl*: needles from EtOH. M.p. 181–3°.

Herzig, Kaserer, *Monatsh.*, 1902, **23**, 577.

Perkin, Simonsen, *J. Chem. Soc.*, 1905, **87**, 863.

### 1 : 1 : 2-Tribromopropane



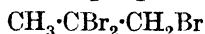
$\text{C}_3\text{H}_5\text{Br}_3$  MW, 281

B.p. 200–1°, 100–3°/20 mm., 83°/6 mm.  $D_4^{20}$  2.356.  $n_D^{20}$  1.573983.

Mereshkowsky, *Ann.*, 1923, **431**, 239.

Bachman, *J. Am. Chem. Soc.*, 1935, **57**, 1090.

### 1 : 2 : 2-Tribromopropane

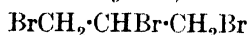


$\text{C}_3\text{H}_5\text{Br}_3$  MW, 281

B.p. 190–1°, 80.6°/20 mm.  $D^{12}$  2.23.  $n_D^{20}$  1.566963.

Mereshkowsky, *Ann.*, 1923, **431**, 241.

**1 : 2 : 3-Tribromopropane** (*Glycerol tribromohydrin, tribromohydrin*)



$\text{C}_3\text{H}_5\text{Br}_3$  MW, 281

M.p. 16–17°. B.p. 219–21°, 115–20°/30 mm., 100–3°/18 mm.  $D_{15}^{18.5}$  2.3955.  $n_D^{18}$  1.584. Zn dust + EtOH  $\rightarrow$  propylene + allyl bromide. Alc. KOH  $\rightarrow$  ethyl propargyl ether. Ag acetate  $\rightarrow$  triacetin.

Johnson, McEwen, *Organic Syntheses*, Collective Vol. I, 507.

### 1 : 1 : 2-Tribromopropionic Acid



$\text{C}_3\text{H}_3\text{O}_2\text{Br}_3$  MW, 311

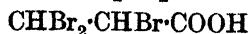
Cryst. from  $\text{CS}_2$ . M.p. 95° (92°). Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Baryta  $\rightarrow$  1 : 2-dibromoacrylic acid.

*Et ester*:  $\text{C}_5\text{H}_7\text{O}_2\text{Br}_3$ . MW, 339. Liq. with aromatic odour. B.p. 140–2°/30 mm.  $D^{23}$  2.084.  $n_D^{23}$  1.532.

Michael, Norton, *Am. Chem. J.*, 1880, **2**, 18.

Berlande, *Bull. soc. chim.*, 1925, **37**, 1390.

### 1 : 2 : 2-Tribromopropionic Acid

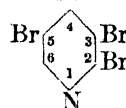


$\text{C}_3\text{H}_3\text{O}_2\text{Br}_3$  MW, 311

Leaflets. M.p. 118°. Very sol. EtOH,  $\text{Et}_2\text{O}$ . Sol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ , ligroin, hot  $\text{H}_2\text{O}$ . Baryta  $\rightarrow$  1 : 2-dibromoacrylic acid.

Hill, Andrews, *Am. Chem. J.*, 1882, **4**, 180.

### 2 : 3 : 5-Tribromopyridine



$\text{C}_5\text{H}_2\text{NBr}_3$  MW, 316

Needles from EtOH.Aq. M.p. 46°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Volatile in steam. Feebly basic.

$\text{B}_2\text{H}_2\text{HgCl}_2$ : cryst. from EtOH.Aq. M.p. 181–2°.

Fischer, Chur, *J. prakt. Chem.*, 1916, **93**, 372.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 940.

### 2 : 3 : 6-Tribromopyridine.

M.p. 82°.

See last reference above.

### 2 : 4 : 6-Tribromopyridine.

Leaflets from EtOH. M.p. 107°.

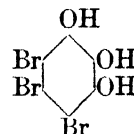
Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 946.

### 3 : 4 : 5-Tribromopyridine.

Cryst. from EtOH. M.p. 106–7°.

Hertog, Wibaut, *Rec. trav. chim.*, 1932, **51**, 950.

### Tribromopyrogallol



$\text{C}_6\text{H}_3\text{O}_3\text{Br}_3$  MW, 363

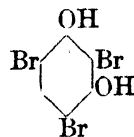
Reddish or brownish needles or leaflets +  $1\text{H}_2\text{O}$  from EtOH.Aq. M.p. 168–70°. Very sol. EtOH. Sol. hot  $\text{H}_2\text{O}$ .

*Tri-Me ether*:  $\text{C}_9\text{H}_9\text{O}_3\text{Br}_3$ . MW, 405. Cryst. M.p. 81.5°.

*Tri-Et ether*:  $\text{C}_{12}\text{H}_{15}\text{O}_3\text{Br}_3$ . MW, 447. M.p. 38–9°.

Moore, Thomas, *J. Am. Chem. Soc.*, 1917, **39**, 987.

### 2 : 4 : 6-Tribromoresorcinol



$\text{C}_6\text{H}_3\text{O}_2\text{Br}_3$  MW, 347

Needles from  $\text{H}_2\text{O}$ . M.p. 112°. Sol. EtOH. Spar. sol. cold  $\text{H}_2\text{O}$ . Gives reactions of quinones.

*Me ether* :  $C_7H_5O_2Br_3$ . MW, 361. Needles. M.p.  $104^\circ$  ( $99^\circ$ ). Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin.

*Di-Me ether* :  $C_8H_7O_2Br_3$ . MW, 375. Cryst. from EtOH.Aq. M.p.  $68-9^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ , ligroin. Insol.  $H_2O$ .

*Me-Et ether* :  $C_9H_9O_2Br_3$ . MW, 389. Needles from EtOH.Aq. M.p.  $75^\circ$ .

*Di-Et ether* :  $C_{10}H_{11}O_2Br_3$ . MW, 403. M.p.  $68-9^\circ$ .

*Monoacetyl* : cryst. from  $CS_2$ . M.p.  $114^\circ$ .

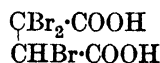
*Diacetyl* : needles. M.p.  $108^\circ$ .

*Monobenzoyl* : prisms from  $CHCl_3$ -pet. ether. M.p.  $120^\circ$ .

Benedikt, *Monatsh.*, 1883, 4, 227.

Raiford, Heyl, *Am. Chem. J.*, 1910, 44, 215.

### Tribromosuccinic Acid



$C_4H_3O_4Br_3$  MW, 355

Plates from  $Et_2O-C_6H_6$ . M.p.  $136^\circ$ . Very sol.  $H_2O$ , EtOH,  $Et_2O$ . Mod. sol.  $C_6H_6$ . Very spar. sol.  $CS_2$ , ligroin. Hot  $H_2O \rightarrow$  2 : 2-dibromoacrylic acid.

Lossen, Bergau, *Ann.*, 1906, 348, 265.

### 2 : 3 : 4-Tribromotoluene



$C_7H_5Br_3$  MW, 329

Cryst. from AcOH. M.p.  $45-6^\circ$ .  $D^{20}_D$  2.456.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 510.

### 2 : 3 : 5-Tribromotoluene.

Cryst. from  $Et_2O$ -toluene. M.p.  $53-4^\circ$ .  $D^{17}_D$  2.467.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 511, 520.

### 2 : 3 : 6-Tribromotoluene.

Leaflets from ligroin or  $CHCl_3$ . M.p.  $58-9^\circ$  ( $60-5^\circ$ ).  $D^{17}_D$  2.471.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 513, 519.

### 2 : 4 : 5-Tribromotoluene.

Needles from EtOH. M.p.  $112-13^\circ$ .  $D^{17}_D$  2.472.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 515, 520.

### 2 : 4 : 6-Tribromotoluene.

Needles from  $Et_2O-AcOEt$ . M.p.  $70^\circ$  ( $65-6^\circ$ ).  $D^{17}_D$  2.479. Very spar. sol. EtOH.

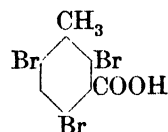
Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 515.

### 3 : 4 : 5-Tribromotoluene.

Needles from  $Et_2O-EtOH$ . M.p.  $91^\circ$  ( $88-9^\circ$ ).  $D^{17}_D$  2.429.

Asinger, *J. prakt. Chem.*, 1935, 142, 297.

### 2 : 4 : 6-Tribromo-*m*-toluic Acid



$C_8H_5O_2Br_3$  MW, 373

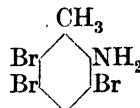
Needles from 50% MeOH. M.p.  $187-188.5^\circ$  after sintering at  $160^\circ$ .

*Amide* :  $C_8H_4ONBr_3$ . MW, 372. Cryst. from 50% EtOH. M.p.  $199-200^\circ$ .

*Nitrile* :  $C_8H_4NBr_3$ . MW, 354. Cryst. from pet. ether or cyclohexane. M.p.  $122-3^\circ$ .

Weissberger, Bach, Strasser, *J. Chem. Soc.*, 1935, 70.

### 3 : 5 : 6-Tribromo-*o*-toluidine



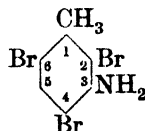
$C_7H_6NBr_3$  MW, 344

M.p.  $87^\circ$ . Sol.  $Et_2O$ ,  $C_6H_6$ , hot EtOH. Mod. sol. pet. ether.

*N-Acetyl* : 3 : 5 : 6-tribromoacet-*o*-toluidide. M.p.  $218^\circ$ .

Blanksma, *Chem. Zentr.*, 1914, I, 971.

### 2 : 4 : 6-Tribromo-*m*-toluidine



$C_7H_6NBr_3$  MW, 344

Needles from EtOH. M.p.  $101^\circ$ . Spar. sol. EtOH.

*N-Acetyl* : 2 : 4 : 6-tribromoacet-*m*-toluidide. Cryst. from EtOH. M.p.  $205^\circ$  ( $155^\circ$ ).

*N-Diacetyl* : m.p.  $103^\circ$ .

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 515.

Fuehs, *Monatsh.*, 1915, 36, 132.

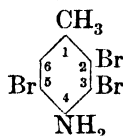
Bureš, Balada, *Chem. Zentr.*, 1927, II, 1345.

**2 : 5 : 6-Tribromo-*m*-toluidine.**

M.p. 93–4°.

N-Acetyl : 2 : 5 : 6-tribromoacet-*m*-toluidide.  
M.p. 179–81°.Neville, Winther, *Ber.*, 1880, 13, 974.**4 : 5 : 6-Tribromo-*m*-toluidine.**

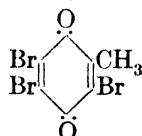
M.p. 96–96.8°.

N-Acetyl : 4 : 5 : 6-tribromoacet-*m*-toluidide.  
Needles from EtOH. M.p. 171–3°.Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 510.**2 : 3 : 5-Tribromo-*p*-toluidine** $C_7H_6NBr_3$  MW, 344

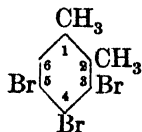
Needles from EtOH. M.p. 82.5–83°.

Neville, Winther, *Ber.*, 1881, 14, 418.**2 : 3 : 6-Tribromo-*p*-toluidine.**

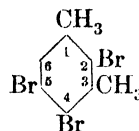
Needles from EtOH. M.p. 118°. Volatile in steam.

Cohen, Dutt, *J. Chem. Soc.*, 1914, 105, 514.**3 : 5 : 6-Tribromotoluquinone (3 : 5 : 6-Tribromo-2-methylbenzoquinone)** $C_7H_3O_2Br_3$  MW, 359Yellow plates from EtOH. M.p. 235–6°.  
Sol.  $Et_2O$ ,  $C_6H_6$ . Very spar. sol. cold EtOH.  
Insol.  $H_2O$ .Fichter, Rinderspacher, *Helv. Chim. Acta*, 1927, 10, 41.Zincke, Klostermann, *Ber.*, 1907, 40, 679.**Tribromoveratrol.**

See under Tribromocatechol.

**3 : 4 : 5-Tribromo-*o*-xylene** $C_8H_7Br_3$  MW, 343Needles from  $Me_2CO$ . M.p. 105°.Jaeger, Blanksma, *Rec. trav. chim.*, 1906, 25, 354.**3 : 4 : 6-Tribromo-*o*-xylene.**Needles. M.p. 86°. Sol.  $Et_2O$ ,  $C_6H_6$ . Spar. sol. cold EtOH, cold  $Me_2CO$ .

See previous reference.

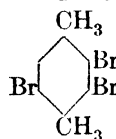
**2 : 4 : 5-Tribromo-*m*-xylene** $C_8H_7Br_3$  MW, 343

Cryst. from EtOH. M.p. 87°.

Jaeger, Blanksma, *Rec. trav. chim.*, 1906, 25, 361.**2 : 4 : 6-Tribromo-*m*-xylene.**Prisms from  $Et_2O-C_6H_6$ . M.p. 85°.Jaeger, Blanksma, *Rec. trav. chim.*, 1906, 25, 357.**4 : 5 : 6-Tribromo-*m*-xylene.**

Cryst. M.p. 105°.

See previous reference.

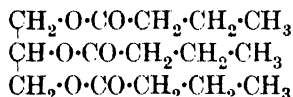
**2 : 3 : 5-Tribromo-*p*-xylene** $C_8H_7Br_3$  MW, 343M.p. 89°. Sol.  $Et_2O$ . Spar. sol.  $Me_2CO$ , cold EtOH.Jaeger, Blanksma, *Rec. trav. chim.*, 1906, 25, 362.**Tributylamine** $C_{12}H_{27}N$  MW, 185B.p. 216.5°.  $D_{20}^{20}$  0.7782. $B, HFeCl_4$  : yellow needles. M.p. 171°.*Butyl iodide* : tetrabutylammonium iodide.  
M.p. 144–5°. Sol.  $H_2O$ , EtOH.*Picrate* : m.p. 105–6°.*Butyl picrate* : tetrabutylammonium picrate.  
M.p. 90–3°.*Benzyl chloride* : tributylbenzylammonium chloride. M.p. 185°.I.G., F.P., 685,345, (*Chem. Abstracts*, 1930, 24, 5765).Skita, Keil, *Monatsh.*, 1929, 53 and 54, 759.Lieben, Rossi, *Ann.* 1871, 158, 172.

**Tributylbenzylammonium chloride.**

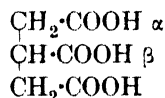
See under Tributylamine.

**Tributyl phosphate.**

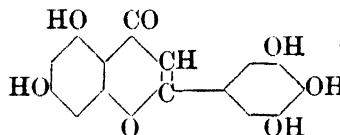
See under Phosphoric Acid.

**Tributylin** (*Glycerol tri-n-butyrate*) $\text{C}_{15}\text{H}_{26}\text{O}_6$ 

MW, 302

Colourless liq. with bitter taste. B.p. 305–10° (287–8°), 190°/15 mm.  $n_D^{20}$  1.43587.  $D_4^{21}$  1.027.Weatherby, McIlvaine, Matlin, *J. Am. Chem. Soc.*, 1925, **47**, 2249.**Tricaprin.**See under *n*-Capric Acid.**Tricaproin.**See under *n*-Caproic Acid.**Tricaprylin.**See under *n*-Caprylic Acid.**Tricarballic Acid** (*Propane-1 : 2 : 3-tricarboxylic acid*) $\text{C}_6\text{H}_8\text{O}_6$ 

MW, 176

Prisms from  $\text{H}_2\text{O}$  or  $\text{Et}_2\text{O}$ . M.p. 166°. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Mod. sol.  $\text{Et}_2\text{O}$ .  $k = 2.2 \times 10^{-4}$  at 25°. $\alpha$ -Me ester:  $\text{C}_7\text{H}_{10}\text{O}_6$ . MW, 190. Oil.  $k = 7.5 \times 10^{-5}$  at 25°. $\beta$ -Me ester: oil.  $k = 9.35 \times 10^{-5}$  at 25°.*Tri-Me ester*:  $\text{C}_9\text{H}_{14}\text{O}_6$ . MW, 218. B.p. 205–8°/48 mm., 150°/13 mm.  $D_4^{20}$  1.18221.  $n_D^{18}$  1.4398.*Tri-Et ester*:  $\text{C}_{12}\text{H}_{20}\text{O}_6$ . MW, 260. Oil. B.p. 295–305°, 180–5°/20 mm. Spar. sol.  $\text{H}_2\text{O}$ .*Triallyl ester*:  $\text{C}_{15}\text{H}_{20}\text{O}_6$ . MW, 296. B.p. 215°/24 mm.  $D_4^{18.4}$  1.0953.  $n_D^{15.9}$  1.46747.*Trichloride*:  $\text{C}_6\text{H}_5\text{O}_3\text{Cl}_3$ . MW, 231.5. Liq. B.p. 140°/14 mm.*Triamide*:  $\text{C}_6\text{H}_{11}\text{O}_3\text{N}_3$ . MW, 173. Prisms from  $\text{H}_2\text{O}$ . M.p. 205–7° decomp. Sol.  $\text{H}_2\text{O}$ . Insol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .*Trinitrile*: 1 : 2 : 3-tricyanopropane,  $\text{C}_3\text{H}_3\text{N}_3$ . MW, 119. Needles from  $\text{Et}_2\text{O}$ . M.p. 47°. Sol. hot  $\text{H}_2\text{O}$ . Spar. sol.  $\text{Et}_2\text{O}$ , cold  $\text{EtOH}$ .*Trihydrazide*: cryst. from  $\text{EtOH.Aq.}$  M.p. 135–6°.  $B, 3\text{HCl}$ : m.p. 148° decomp.*Trianilide*: needles from  $\text{PhNO}_2$ . M.p. 252°. $\alpha\beta$ -Anhydride:  $\text{C}_6\text{H}_6\text{O}_5$ . MW, 158. Needles from  $\text{AcOH-CHCl}_3$ . M.p. 131–2°. B.p. 215–25°/45 mm. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{AcOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . $\alpha\beta$ -Imide:  $\text{C}_6\text{H}_7\text{O}_4\text{N}$ . MW, 157. Prisms from  $\text{C}_6\text{H}_6$ . M.p. 127–8°. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Amide: cryst. from  $\text{EtOH.Aq.}$  M.p. 173°. $\alpha\beta$ -Anil:  $\text{C}_{12}\text{H}_{11}\text{O}_4\text{N}$ . MW, 233. Plates from  $\text{H}_2\text{O}$ . M.p. 137°. Me ester:  $\text{C}_{13}\text{H}_{13}\text{O}_4\text{N}$ . MW, 247. Needles from  $\text{Et}_2\text{O-EtOH}$ . M.p. 106°. Et ester:  $\text{C}_{14}\text{H}_{15}\text{O}_4\text{N}$ . MW, 261. Needles. M.p. 90°. Anilide: needles from  $\text{EtOH.Aq.}$  M.p. 168°.Clarke, Murray, *Organic Syntheses*, Collective Vol. I, 508.Bertram, *Ber.*, 1905, **38**, 1620.Bone, Sprankling, *J. Chem. Soc.*, 1902, **81**, 34.Emery, *Ber.*, 1891, **24**, 597; 1889, **22**, 2920.**Tricetin** (5 : 7 : 3' : 4' : 5' - Pentahydroxy-flavone) $\text{C}_{15}\text{H}_{10}\text{O}_7$ 

MW, 302

Needles +  $\text{H}_2\text{O}$  from  $\text{EtOH.Aq.}$  Decomp. above 330°. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol.  $\text{NaOH} \rightarrow$  orange-red sol. Alc.  $\text{FeCl}_3 \rightarrow$  reddish-brown col.  $\rightarrow$  olive-green.

3' : 5'-Di-Me ether: see Tricin.

3' : 4' : 5'-Tri-Me ether:  $\text{C}_{18}\text{H}_{16}\text{O}_7$ . MW, 344. Yellow needles from  $\text{AcOH}$ . M.p. 269–70°. Conc.  $\text{H}_2\text{SO}_4$  and  $\text{NaOH}$  give yellow sols. Alc.  $\text{FeCl}_3 \rightarrow$  reddish-brown to olive-green col. Diacetyl: needles from  $\text{EtOH}$ . M.p. 160–2°.Penta-Me ether:  $\text{C}_{20}\text{H}_{20}\text{O}_7$ . MW, 372. Needles from  $\text{MeOH}$ . M.p. 192–3°.

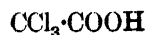
Tetra-acetyl: needles. M.p. 260–1°.

Penta-acetyl: needles from  $\text{EtOH-AcOH}$ . M.p. 244° (241–2°).Anderson, *Chem. Zentr.*, 1932, II, 3899; 1933, II, 2012.**Trichloroacetaldehyde.**

See Chloral.

**Trichloroacetanilide.**

See under Trichloroaniline.

**Trichloroacetic Acid** $\text{C}_2\text{HO}_2\text{Cl}_3$ 

MW, 163.5

Deliquescent cryst. M.p. 58°. B.p. 196.5°, 141–2°/25 mm.  $D_4^{20}$  1.6237. Sol.  $\text{H}_2\text{O}$ .  $k = 1.2$  approx. Hot  $\text{H}_2\text{O}$  or dil. alkalis  $\rightarrow$  chloroform +  $\text{CO}_2$ . Conc. alkalis  $\rightarrow$  formic acid.

*Me ester*:  $C_3H_5O_2Cl_3$ . MW, 177.5. B.p. 152°, 52-4°/12 mm.

*Et ester*:  $C_4H_7O_2Cl_3$ . MW, 191.5. B.p. 167.1°/754.8 mm., 60-1°/12 mm.  $D_{15}^{20}$  1.3886.  $n_D^{20}$  1.45068.

*Propyl ester*:  $C_5H_9O_2Cl_3$ . MW, 205.5. B.p. 187°.

*Isobutyl ester*:  $C_6H_{11}O_2Cl_3$ . MW, 219.5. B.p. 187-9°.

active-*Amyl ester*:  $C_7H_{13}O_2Cl_3$ . MW, 233.5. B.p. 210-12°/721 mm.  $D_4^{22}$  1.233.  $n_D^{21}$  1.4517.  $[\alpha]_D^{22} + 3.54^\circ$ .

*Allyl ester*:  $C_5H_5O_2Cl_3$ . MW, 203.5. B.p. 183-4°/766 mm.

*Vinyl ester*:  $C_4H_3O_2Cl_3$ . MW, 189.5. B.p. 149°.

*Phenyl ester*:  $C_8H_5O_2Cl_3$ . MW, 239.5. B.p. 254-5° decomp.

2-Naphthyl ester: m.p. 86-7°.

*Benzyl ester*: viscous oil. B.p. 178.5°/50 mm.  $D_4^{21}$  1.3887.  $n_D^{18}$  1.5288.

*Anhydride*:  $C_4O_3Cl_6$ . MW, 309. B.p. 222-4° decomp., 140°/110 mm.  $D^{20}$  1.6908.

*Chloride*:  $C_2CCl_4$ . MW, 182. B.p. 118°  $D_4^{20}$  1.6564.

*Bromide*:  $C_2OCl_3Br$ . MW, 226.5. B.p. 143°.  $D_{15}^{15}$  1.900.

*Iodide*:  $C_2OCl_3I$ . MW, 273.5. B.p. about 180°.

*Amide*:  $C_3H_2ONCl_3$ . MW, 162.5. Cryst. from  $H_2O$ . M.p. 141°. B.p. 238-9°/746 mm. Sol. EtOH, Et<sub>2</sub>O. Spar. sol.  $H_2O$ . Sublimes in plates.

*Methylamide*: cryst. from Et<sub>2</sub>O. M.p. 105-6°.

*Dimethylamide*: b.p. 230-3° slight decomp.  $D_{15}^{15}$  1.441.

*Ethylamide*: cryst. M.p. 74°. B.p. 229-30° slight decomp.

*Diethylamide*: prisms with odour of peppermint. M.p. 27°.

*Nitrile*:  $C_2NCl_3$ . MW, 144.5. B.p. 83-4°.  $D_{12}^{12}$  1.439.

*Imide*: di-trichloroacetamide.  $C_4HO_3NCl_6$ . MW, 308. Cryst. from pet. ether. M.p. 86°.

*Anilide*: cryst. from EtOH.Aq. M.p. 95-7°.

*o-Nitroanilide*: yellow needles from EtOH. M.p. 70-2°.

*p-Nitroanilide*: needles from EtOH. M.p. 146-7°.

Anschütz, Haslam, *Ann.*, 1889, 253, 124.

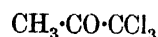
Judson, *Ber.*, 1870, 3, 782.

Clermont, *Ann. chim. phys.*, 1885, 6, 135.

### Trichloro-acetoisopropyl Alcohol.

See Chloralacetone.

### 1 : 1 : 1-Trichloroacetone



$C_3H_3OCl_3$  MW, 161.5

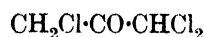
Liq. with odour of camphor. B.p. 149°/764 mm. (134°), 57°/48 mm.

*Semicarbazone*: needles. M.p. 140° decomp.

Blaise, *Compt. rend.*, 1912, 155, 1253.

Schlotterbeck, *Ber.*, 1909, 42, 2561.

### 1 : 1 : 3-Trichloroacetone



$C_3H_3OCl_3$  MW, 161.5

B.p. 172°.

Cloëz, *Ann. chim. phys.*, 1886, 9, 176.

### Trichloro-acet-toluidide.

See under Trichlorotoluidine.

### Trichloroacrylic Acid



$C_3HO_2Cl_3$  MW, 175.5

Prisms from CS<sub>2</sub>. M.p. 76°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, hot  $H_2O$ . Spar. sol. cold  $H_2O$ .  $k$  = about  $5.4 \times 10^{-2}$  at 25°.

*Et ester*:  $C_5H_5O_2Cl_3$ . MW, 203.5. Liq. with odour of peppermint. B.p. 192-4°, 112-14°/50 mm.  $D_4^{20}$  1.3740.  $n_D^{20}$  1.4839.

*Anhydride*:  $C_6O_3Cl_6$ . MW, 333. Cryst. M.p. 39-40°. Insol.  $H_2O$ .

*Chloride*:  $C_3OCl_4$ . MW, 194. B.p. 158°.  $n_D^{18}$  1.52709.

*Amide*:  $C_3H_2ONCl_3$ . MW, 174.5. Needles. M.p. 96-7°. Sol. EtOH, Et<sub>2</sub>O, hot  $H_2O$ . Spar. sol. cold  $H_2O$ .

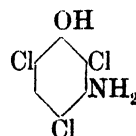
*Nitrile*:  $C_3NCl_3$ . MW, 156.5. Cryst. M.p. 20°.  $n_D^{20}$  1.5100.

Prins, D.R.P., 261,689, (*Chem. Zentr.*, 1913, II, 394).

Fritsch, *Ann.*, 1897, 297, 315.

Mabery, *Am. Chem. J.*, 1887, 9, 3.

### 2 : 4 : 6-Trichloro-*m*-aminophenol



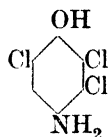
$C_6H_4ONCl_3$  MW, 212.5

Leaflets from ligroin. M.p. 95-6°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>,  $C_6H_6$ . Spar. sol.  $H_2O$ . Aq. sol.  $\rightarrow$  violet-red col. with FeCl<sub>3</sub>.

*N-Acetyl*: needles from toluene. M.p. 185-186.5°. FeCl<sub>3</sub>  $\rightarrow$  violet col.

Jacobs, Heidelberger, Rolf, *J. Am. Chem. Soc.*, 1919, 41, 463.

Daccomo, *Ber.*, 1885, 18, 1166.

**2 : 3 : 6-Trichloro-*p*-aminophenol** $C_6H_4ONCl_3$ 

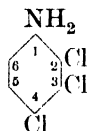
MW, 212.5

Needles from EtOH. M.p. 159° → a brown liq. Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O.

N-2 : 4-Dinitrophenyl : orange-red cryst. M.p. 211°. Acetyl : yellow prisms. M.p. 153°.

Kohn, Fink, *Monatsh.*, 1930, **56**, 138.

Schmitt, Andresen, *J. prakt. Chem.*, 1881, **24**, 426.

**2 : 3 : 4-Trichloroaniline** $C_6H_4NCl_3$ 

MW, 196.5

Needles from ligroin. M.p. 67.5°. B.p. 292°/774 mm. Sol. EtOH.

N-Acetyl : 2 : 3 : 4-trichloroacetanilide. Needles. M.p. 123°.

Zincke, Kuchenbecker, *Ann.*, 1904, **330**, 56.

Beilstein, Kurbatow, *Ann.*, 1879, **196**, 233.

**2 : 3 : 5-Trichloroaniline.**

Needles from pet. ether. M.p. 73°.

Hodgson, Kershaw, *J. Chem. Soc.*, 1929, 2921.

**2 : 3 : 6-Trichloroaniline.**

Cryst. from EtOH. M.p. 63-4°.

N-Acetyl : 2 : 3 : 6-trichloroacetanilide. Cryst. from EtOH.Aq. M.p. 172-3° (134.5°).

Hüffer, *Rec. trav. chim.*, 1921, **40**, 457.

Chattaway, Orton, Hurtley, *J. Chem. Soc.*, 1900, **77**, 802.

**2 : 4 : 5-Trichloroaniline.**

Needles from ligroin. M.p. 96.5°. B.p. about 270°. Sol. EtOH, CS<sub>2</sub>. Spar. sol. ligroin. Volatile in steam.

N-Acetyl : 2 : 4 : 5-trichloroacetanilide. Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 190°.

Dyson, George, Hunter, *J. Chem. Soc.*, 1926, 3044.

Beilstein, Kurbatow, *Ann.*, 1879, **196**, 233.

**2 : 4 : 6-Trichloroaniline.**

Needles from ligroin. M.p. 78.5°. B.p. 262°/746 mm. Sol. EtOH, Et<sub>2</sub>O, CS<sub>2</sub>, ligroin.

N-Me : 2 : 4 : 6-trichloromethylaniline. C<sub>7</sub>H<sub>6</sub>NCl<sub>3</sub>. MW, 210.5. Needles. M.p. 32°. B.p. 260°. N-Acetyl : m.p. 89-90°. N-Benzoyl : m.p. 96-7°.

N-Di-Me : 2 : 4 : 6-trichlorodimethylaniline. C<sub>8</sub>H<sub>8</sub>NCl<sub>3</sub>. MW, 224.5. B.p. 247°/745 mm.

N-Et : 2 : 4 : 6-trichloroethylaniline. C<sub>8</sub>H<sub>8</sub>NCl<sub>3</sub>. MW, 224.5. B.p. 148-53°/25 mm. N-Acetyl : m.p. 50-1°. N-Benzoyl : m.p. 127-8°.

N-Formyl : 2 : 4 : 6-trichloroformanilide. Needles from EtOH. M.p. 180°.

N-Acetyl : 2 : 4 : 6-trichloroacetanilide. Needles. M.p. 207-8° (204°).

N-Diacetyl : prisms from EtOH. M.p. 81-2°.

N-Propionyl : prisms from EtOH. M.p. 161°.

N-Benzoyl : needles from EtOH. M.p. 174°.

Picrate : crimson prisms from EtOH. M.p. 82.5°.

Chattaway, Irving, *J. Chem. Soc.*, 1933, 142.

Beilstein, Kurbatow, *Ann.*, 1879, **196**, 230.

**3 : 4 : 5-Trichloroaniline.**

Needles from EtOH.Aq. M.p. 100° (89°).

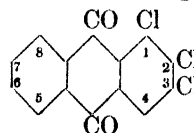
N-Acetyl : 3 : 4 : 5-trichloroacetanilide. M.p. 207-8°.

Dyson, George, Hunter, *J. Chem. Soc.*, 1926, 3043.

Holleman, *Rec. trav. chim.*, 1918, **37**, 196.

**Trichloroanisole.**

See under Trichlorophenol.

**1 : 2 : 3-Trichloroanthraquinone** $C_{14}H_5O_2Cl_3$ 

MW, 311.5

Yellow needles from AcOH. M.p. 194-5°.

Goldberg, *J. Chem. Soc.*, 1931, 1789.

**1 : 2 : 4-Trichloroanthraquinone.**

Yellow cryst. from CHCl<sub>3</sub>. M.p. 185.5°. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. Et<sub>2</sub>O, AcOH. Spar. sol. EtOH.

Graebe, Rostowzew, *Ber.*, 1901, **34**, 2113.

Goldberg, *J. Chem. Soc.*, 1931, 1771.

**1 : 2 : 5-Trichloroanthraquinone.**

Yellow needles from AcOH. M.p. 235-6°.

Goldberg, *J. Chem. Soc.*, 1931, 1792.

Cf. Schilling, *Ber.*, 1913, **46**, 1068.

**1 : 2 : 6-Trichloroanthraquinone.**

Yellow needles from AcOH. M.p. 222–3°.

Goldberg, *J. Chem. Soc.*, 1931, 1784.**1 : 2 : 7-Trichloroanthraquinone.**

Needles from AcOH. M.p. 225–6° (259–60°).

Goldberg, *J. Chem. Soc.*, 1931, 1786.Keimatsu, Hirano, Tanabe, *Chem. Abstracts*, 1929, 23, 4214.**1 : 3 : 6-Trichloroanthraquinone.**

Yellow needles from AcOH. M.p. 212–13°.

Goldberg, *J. Chem. Soc.*, 1931, 1790.**1 : 3 : 7-Trichloroanthraquinone.**

Lemon-yellow needles from AcOH. M.p. 216–17°.

See previous reference.

**1 : 4 : 5-Trichloroanthraquinone.**

Yellow needles from AcOH. M.p. 258° (253–4°).

Badische, D.R.P., 214,714, (*Chem. Zentr.*, 1909, II, 1603).Goldberg, *J. Chem. Soc.*, 1931, 1791.Cf. Schilling, *Ber.*, 1913, 46, 1068.**1 : 4 : 6-Trichloroanthraquinone.**

Yellow needles from AcOH. M.p. 238°.

Goldberg, *J. Chem. Soc.*, 1931, 1784.Jaroschy, *Monatsh.*, 1913, 34, 3.**2 : 3 : 5-Trichloroanthraquinone.**

Yellow needles from AcOH. M.p. 227–8°.

Goldberg, *J. Chem. Soc.*, 1932, 80.**2 : 3 : 6-Trichloroanthraquinone.**

Yellow needles from AcOH. M.p. 245°.

Goldberg, *J. Chem. Soc.*, 1932, 81.**2 : 3 : 4-Trichlorobenzaldehyde** $C_7H_3OCl_3$ 

MW, 209.5

Needles from EtOH. M.p. 90°.

Seelig, *Ann.*, 1887, 237, 149.**2 : 3 : 5-Trichlorobenzaldehyde.**Needles from EtOH. M.p. 56°. Sol. most org. solvents. Volatile in steam.  $KMnO_4 \longrightarrow$  2 : 3 : 5-trichlorobenzoic acid.Hodgson, Beard, *J. Chem. Soc.*, 1927, 2382.**2 : 3 : 6-Trichlorobenzaldehyde.**

Needles from ligroin. M.p. 86–7°.

Geigy, D.R.P., 199,943, (*Chem. Zentr.*, 1908, II, 363).**2 : 4 : 5-Trichlorobenzaldehyde.**Needles. M.p. 112–13°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Volatile in steam. Oxidises in air.Seelig, *Ann.*, 1887, 237, 148.Beilstein, Kuhlberg, *Ann.*, 1869, 152, 238.**2 : 4 : 6-Trichlorobenzaldehyde.**

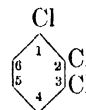
Needles from ligroin. M.p. 58–9°.

Geigy, D.R.P., 199,943, (*Chem. Zentr.*, 1908, II, 363).**3 : 4 : 5-Trichlorobenzaldehyde.**Needles from EtOH. Aq. M.p. 90–1°.  $KMnO_4 \longrightarrow$  3 : 4 : 5-trichlorobenzoic acid.

Phenylhydrazones: yellow cryst. M.p. 147°.

p-Nitrophenylhydrazones: orange needles from  $PhNO_2$ . M.p. 342°.

Semicarbazones: cryst. from EtOH. M.p. 252–4°.

van der Bunt, *Rec. trav. chim.*, 1929, 48, 131.**1 : 2 : 3-Trichlorobenzene** $C_6H_3Cl_3$ 

MW, 181.5

Plates from EtOH. M.p. 53–4°. B.p. 218–19°. Mod. sol. EtOH. Volatile in steam.

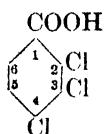
Holleman, *Rec. trav. chim.*, 1918, 37, 196.Beilstein, Kurbatow, *Ann.*, 1878, 192, 234.**1 : 2 : 4-Trichlorobenzene.**M.p. 17°. B.p. 210°.  $D_{20}^{25}$  1.4460.van de Lande, *Rec. trav. chim.*, 1932, 51, 98.Cohen, Hartley, *J. Chem. Soc.*, 1905, 87, 1363.**1 : 3 : 5-Trichlorobenzene.**

Needles. M.p. 63.5°. B.p. 208.5°/763.8 mm.

Holleman, *Rec. trav. chim.*, 1918, 37, 198.Jackson, Lamai, *Am. Chem. J.*, 1896, 18, 667.



## 2 : 3 : 4-Trichlorobenzoic Acid

 $C_7H_3O_2Cl_3$ 

MW, 225.5

Needles from  $H_2O$ . M.p.  $186-7^\circ$ .Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1328.

## 2 : 3 : 5-Trichlorobenzoic Acid.

Needles from  $H_2O$ . M.p.  $163^\circ$ . Sol. common org. solvents. Spar. sol.  $H_2O$ . Non-volatile in steam.*Chloride*:  $C_7H_2OCl_4$ . MW, 244. Cryst. from AcOEt. M.p.  $36^\circ$ .*Amide*:  $C_7H_4ONCl_3$ . MW, 224.5. Needles from AcOH.Aq. M.p.  $204-5^\circ$ .*Nitrile*:  $C_7H_2NCl_3$ . MW, 206.5. Needles from EtOH.Aq. M.p.  $87^\circ$ . Volatile in steam.Hodgson, Beard, *J. Chem. Soc.*, 1927, 2382.Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1331.Matthews, *J. Chem. Soc.*, 1901, **79**, 46.

## 2 : 4 : 5-Trichlorobenzoic Acid.

Needles from  $H_2O$ . M.p.  $162-4^\circ$ . Sol. EtOH. Prac. insol. cold  $H_2O$ . Sublimes in needles.*Et ester*:  $C_9H_7O_2Cl_3$ . MW, 253.5. Needles from EtOH. M.p.  $65^\circ$ . Mod. sol. EtOH.*Chloride*: m.p.  $41^\circ$ . B.p.  $272^\circ$  slight decomp. Sol.  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ .*Amide*: needles from  $C_6H_6$ . M.p.  $167-5^\circ$ . Sol. EtOH. Spar. sol. hot  $H_2O$ . Very spar. sol.  $Et_2O$ ,  $CS_2$ .Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1335.Beilstein, Kuhlberg, *Ann.*, 1869, **152**, 234.

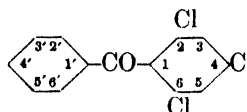
## 2 : 4 : 6-Trichlorobenzoic Acid.

Cryst. from  $H_2O$ . M.p.  $164^\circ$ . Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .*Chloride*: oil. B.p.  $275^\circ$ .*Amide*: cryst. M.p.  $181^\circ$ . Mod. sol. hot  $H_2O$ .*Nitrile*: needles. M.p.  $77-5^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol. hot  $H_2O$ .Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1336.Sudborough, *J. Chem. Soc.*, 1895, **67**, 602.

## 3 : 4 : 5-Trichlorobenzoic Acid.

Needles from EtOH.Aq. M.p.  $210^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $CS_2$ . Prac.insol.  $H_2O$ . Sublimes in needles. Spar. volatile in steam.*Et ester*: needles. M.p.  $86^\circ$ .*Chloride*: prisms. M.p.  $36^\circ$ . Sol.  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ .*Amide*: needles from  $C_6H_6$ . M.p.  $176^\circ$ . Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .van der Bunt, *Rec. trav. chim.*, 1929, **48**, 132.Salkowski, *Ann.*, 1872, **163**, 28.

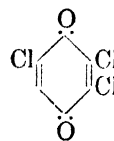
## 2 : 4 : 6-Trichlorobenzophenone

 $C_{13}H_7OCl_3$ 

MW, 285.5

Cryst. from pet. ether. M.p.  $130-5^\circ$ . B.p.  $356^\circ/763$  mm.Montagne, *Rec. trav. chim.*, 1907, **26**, 274.

## 2 : 5 : 2'-Trichlorobenzophenone.

Needles from EtOH. M.p.  $145-7^\circ$ .Ganzmüller, *J. prakt. Chem.*, 1933, **138**, 311.Trichloro-*p*-benzoquinone $C_6HO_2Cl_3$ 

MW, 211.5

Yellow plates from EtOH. M.p.  $169-70^\circ$ . Sol.  $Et_2O$ , hot EtOH. Insol. cold  $H_2O$ . Sublimes.Erdélyi, *Ber.*, 1930, **63**, 1200.v. Knapp, Schultz, *Ann.*, 1881, **210**, 174.

## Trichlorobromomethane (Trichloromethyl bromide)

 $CCl_3Br$  $CCl_3Br$ 

MW, 198.5

M.p.  $-21^\circ$ . B.p.  $104.3^\circ$ .  $D_4^{20}$  2.05496. Photochem. reaction with Cl  $\rightarrow$  carbon tetrachloride. Sensitive to light.Wonders, *Bull. sci. acad. roy. Belg.*, 1934, **20**, 782.Löw, *Z. Chem.*, 1869, 624.

## 1 : 2 : 3-Trichlorobutane

 $CH_3\cdot CHCl\cdot CHCl\cdot CH_2Cl$  $C_4H_7Cl_3$ 

MW, 161.5

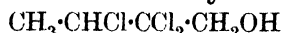
B.p. 165–8°/725 mm., 79–80°/32 mm., 62–3°/28 mm.  $D_0^{20}$  1.3241. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O.

de Montmollin, Matile, *Helv. Chim. Acta*, 1924, 7, 106.

Charon, *Ann. chim. phys.*, 1899, 17, 230.

*Note*.—Losanitsch, *Chem. Zentr.*, 1913, II, 754, described a trichlorobutane of unknown constitution, b.p. 90°/16 mm.

### 2 : 2 : 3-Trichloro-*n*-butyl Alcohol



C<sub>4</sub>H<sub>7</sub>OCl<sub>3</sub> MW, 177.5

Prisms. M.p. 61.5–62°. B.p. 199–200°, 120°/45 mm. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Reduces warm Fehling's.  $\text{HNO}_3 \rightarrow$  1 : 1 : 2-trichlorobutyric acid.

*Phosphate*: needles from EtOH. M.p. 85.3°.

Mering, *Z. physiol. Chem.*, 1882, 6, 493.

Garzarolli-Thurnlakh, Popper, *Ann.*, 1884, 223, 166.

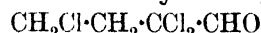
### Trichloro-*tert*.-butyl Alcohol.

*See* Acetone-chloroform.

### 1 : 1 : 2-Trichlorobutyraldehyde.

*See* Butylchloral.

### 1 : 1 : 3-Trichlorobutyraldehyde

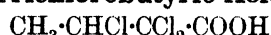


C<sub>4</sub>H<sub>5</sub>OCl<sub>3</sub> MW, 175.5

Liq. Spar. sol. H<sub>2</sub>O. Forms no hydrate. Oxidises slowly in air.  $\text{HNO}_3 \rightarrow$  1 : 1 : 3-trichlorobutyric acid. Forms bisulphite comp.

Natterer, *Monatsh.*, 1883, 4, 551.

### 1 : 1 : 2-Trichlorobutyric Acid



C<sub>4</sub>H<sub>5</sub>O<sub>2</sub>Cl<sub>3</sub> MW, 191.5

Plates or needles from pet. ether. M.p. 60°. B.p. 236–8°.  $k =$  about 0.18 at 18°.

*Ester*: C<sub>6</sub>H<sub>5</sub>O<sub>2</sub>Cl<sub>3</sub>. MW, 219.5. B.p. 212°, 101.5°/17 mm.  $D_0^{20}$  1.3138.

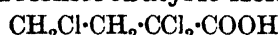
*Chloride*: C<sub>4</sub>H<sub>4</sub>OCl<sub>4</sub>. MW, 210. B.p. 162–6°.

*Amide*: C<sub>4</sub>H<sub>6</sub>ONCl<sub>3</sub>. MW, 190.5. Plates from EtOH. M.p. 96°. Spar. sol. H<sub>2</sub>O.

Krämer, Pinner, *Ber.*, 1870, 3, 389.

Valentin, *Ber.*, 1895, 28, 2661.

### 1 : 1 : 3-Trichlorobutyric Acid

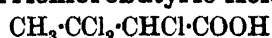


C<sub>4</sub>H<sub>5</sub>O<sub>2</sub>Cl<sub>3</sub> MW, 191.5

Cryst. M.p. 73–5°. Sol. 20 parts H<sub>2</sub>O.

Natterer, *Monatsh.*, 1883, 4, 551.

### 1 : 2 : 2-Trichlorobutyric Acid



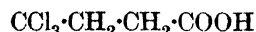
C<sub>4</sub>H<sub>5</sub>O<sub>2</sub>Cl<sub>3</sub> MW, 191.5

Dict. of Org. Comp.—III.

Plates from ligroin. M.p. 51.5–52°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

Szenic, Taggesell, *Ber.*, 1895, 28, 2665.

### 3 : 3 : 3-Trichlorobutyric Acid



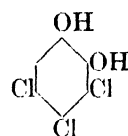
C<sub>4</sub>H<sub>5</sub>O<sub>2</sub>Cl<sub>3</sub> MW, 191.5

M.p. 57°.

*Anhydride*: C<sub>8</sub>H<sub>4</sub>O<sub>3</sub>Cl<sub>6</sub>. MW, 365. B.p. 138–40°/20 mm.  $D_0^{20}$  0.970.

Baroni, *Gazz. chim. ital.*, 1933, 63, 23.

### 3 : 4 : 5-Trichlorocatechol



C<sub>6</sub>H<sub>3</sub>O<sub>2</sub>Cl<sub>3</sub> MW, 213.5

Two hydrates are known:

(i) + 1H<sub>2</sub>O. Prisms from AcOH. M.p. 115° (104–5°). Sol. EtOH, Et<sub>2</sub>O, AcOH. Insol. cold H<sub>2</sub>O.  $\text{FeCl}_3 \rightarrow$  green col.

(ii) +  $\frac{1}{2}$ H<sub>2</sub>O. Prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 134–5°.

1(or 2)-*Me ether*: trichloroguaiacol. C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>Cl<sub>3</sub>. MW, 227.5. Needles from pet. ether. M.p. 107–8°. Volatile in steam. *Benzoyl*: cryst. from ligroin. M.p. 128–9°.

2(or 1)-*Me ether*: needles. M.p. 114–15°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Volatile in steam.  $\text{FeCl}_3 \rightarrow$  green col.

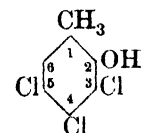
*Di-Me ether*: 3 : 4 : 5-trichloroveratrol. C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>Cl<sub>3</sub>. MW, 241.5. Needles. M.p. 68–9°. Sol. C<sub>6</sub>H<sub>6</sub>, hot EtOH. Insol. H<sub>2</sub>O.

Cousin, *Ann. chim. phys.*, 1903, 29, 60, 90.

Jackson, Boswell, *Am. Chem. J.*, 1906, 35, 526.

Peratoner, Ortoleva, *Gazz. chim. ital.*, 1898, 28, 230.

### 3 : 4 : 5-Trichloro-*o*-cresol



C<sub>7</sub>H<sub>5</sub>OCl<sub>3</sub> MW, 211.5

Needles from pet. ether. M.p. 77°. Sol. common org. solvents.

*Acetyl*: needles from MeOH.Aq. M.p. 45°.

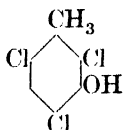
Zincke, Preiss, *Ann.*, 1918, 417, 204.

**3 : 5 : 6-Trichloro-*o*-cresol.**

Needles from AcOH. M.p. 62°. Sol. common org. solvents except pet. ether.

*Benzoyl* : needles from EtOH. M.p. 110°.

Zincke, Preiss, *Ann.*, 1918, **417**, 205.

**2 : 4 : 6-Trichloro-*m*-cresol**

$C_7H_5OCl_3$

MW, 211.5

Needles or plates from  $H_2O$ . M.p. 47°. B.p. 265°, 142–4°/14 mm. Sol. common org. solvents. Spar. sol.  $H_2O$ . Volatile in steam. Complex with cuprammonium sulphate employed as a fungicide.

*Me ether* :  $C_8H_7OCl_3$ . MW, 225.5. Needles from AcOH.Aq. M.p. 54–5° (46°).

*Et ether* :  $C_9H_9OCl_3$ . MW, 239.5. M.p. 35.5°.

*Phenyl ether* :  $C_{13}H_9OCl_3$ . MW, 287.5. M.p. 103°.

*Acetyl* : cryst. from  $Et_2O$ . M.p. 35° (32°). B.p. 273–4°.

*Benzoyl* : plates. M.p. 53°.

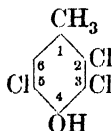
*Benzenesulphonyl* : plates. M.p. 121°.

*p-Toluenesulphonyl* : plates. M.p. 92–3°.

Huston, Chen, *J. Am. Chem. Soc.*, 1933, **56**, 4218.

Crowther, McCombie, *J. Chem. Soc.*, 1913, **103**, 542.

Raiford, *Am. Chem. J.*, 1911, **46**, 423.

**2 : 3 : 5-Trichloro-*p*-cresol**

$C_7H_5OCl_3$

MW, 211.5

Needles from AcOH or pet. ether. M.p. 66–7°.

*Acetyl* : needles from AcOH.Aq. M.p. 37–8°.

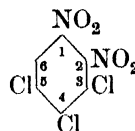
*Benzoyl* : plates from EtOH.Aq. M.p. 89°.

Zincke, *Ann.*, 1903, **328**, 279.

**2 : 3 : 6-Trichloro-*p*-cresol.**

Cryst. from AcOH. M.p. 85–6°.

Datta, Mitter, *J. Am. Chem. Soc.*, 1919, **41**, 2034.

**3 : 4 : 5-Trichloro-*o*-dinitrobenzene**

$C_6H_3O_4N_2Cl_3$

MW, 271.5

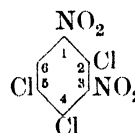
Yellow cryst. from EtOH. M.p. 105–6°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 451.

**3 : 4 : 6-Trichloro-*o*-dinitrobenzene.**

Yellow prisms. M.p. 70–1°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 453.

**2 : 4 : 5-Trichloro-*m*-dinitrobenzene**

$C_6H_3O_4N_2Cl_3$

MW, 271.5

Pale yellow prisms from EtOH. M.p. 103.5°. B.p. 335°.  $D^{25}_D$  1.850. Sol.  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ , hot EtOH.

Qvist, Salo, *Chem. Zentr.*, 1936, **I**, 540.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 452.

**2 : 4 : 6-Trichloro-*m*-dinitrobenzene.**

Prisms from EtOH. M.p. 129.5°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ .

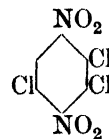
Jackson, Wing, *Am. Chem. J.*, 1887, **9**, 353.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 451.

**4 : 5 : 6-Trichloro-*m*-dinitrobenzene.**

Greenish-yellow needles from EtOH. M.p. 94°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 452.

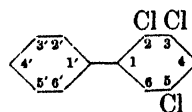
**2 : 3 : 5-Trichloro-*p*-dinitrobenzene**

$C_6H_3O_4N_2Cl_3$

MW, 271.5

Yellow needles from EtOH. M.p. 102.5°.

Hüffer, *Rec. trav. chim.*, 1921, **40**, 457.

**2 : 3 : 5-Trichlorodiphenyl**

$C_{12}H_7Cl_3$

MW, 257.5

Needles from EtOH.Aq. M.p. 41°.

Hinkel, Hey, *J. Chem. Soc.*, 1928, 2790.

**2 : 5 : 4'-Trichlorodiphenyl.**

M.p. 67°.

Bellavita, *Gazz. chim. ital.*, 1935, **65**, 632.**3 : 4 : 2'-Trichlorodiphenyl.**

M.p. 65-6° (54°).

See previous reference and also

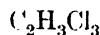
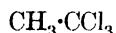
Mascarelli, Gatti, Longo, *Gazz. chim. ital.*, 1933, **63**, 654.**3 : 5 : 2'-Trichlorodiphenyl.**

Pale yellow needles from EtOH. M.p. 58°.

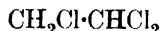
Hinkel, Hey, *J. Chem. Soc.*, 1928, 2791.**3 : 5 : 4'-Trichlorodiphenyl.**

Needles from EtOH. M.p. 88°.

See previous reference.

**1 : 1 : 1-Trichloroethane (Methylchloroform)**

MW, 133.5

B.p. 74.9°/758 mm.  $D_{25}^{25}$  1.31144.  $n_D^{21}$  1.419861.Städel, *Ann.*, 1879, **195**, 184.I.G., D.R.P., 523,436, (*Chem. Zentr.*, 1931, I, 3607).**1 : 1 : 2-Trichloroethane**

MW, 133.5

F.p. - 35.5°. B.p. 113.7°.  $D_4^{20}$  1.4416.Prins, *Rec. trav. chim.*, 1926, **45**, 80.**2 : 2 : 2-Trichloroethyl Alcohol**

MW, 149.5

Plates. M.p. 19°. B.p. 151°, 94-7°/125 mm. Very hygroscopic. Misc. in all proportions with EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O. Sol. aq. KOH. Reduces warm Fehling's. Fuming HNO<sub>3</sub> → trichloroacetic acid.  $\text{PCl}_5 \rightarrow$  2 : 2 : 2-trichloroethyl phosphite.  $\text{PCl}_5 \rightarrow$  2 : 2 : 2-trichloroethyl phosphate.

p-Nitrobenzoyl : prisms from EtOH. M.p. 71°.

p-Aminobenzoyl : needles from pet. ether. M.p. 87°.

Urethane : needles. M.p. 64-5°.

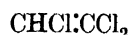
1-Naphthylurethane : cryst. from ligroin. M.p. 120°.

Phosphite : oil. B.p. 263°.

Phosphate : cryst. M.p. 73-4°. Sol. Et<sub>2</sub>O. Spar. sol. ligroin.

Chalmers, *Organic Syntheses*, 1935, XV, 80.

Dean, Wolf, *J. Am. Chem. Soc.*, 1936, **58**, 332.

**Trichloroethylene**

MW, 131.5

F.p. - 86.4°. M.p. - 73°. B.p. 88-90°, 25°/73 mm.  $D_{15}^{15}$  1.4397.  $n_D^{20}$  1.47820. Non-inflammable and employed as a germicide and industrial solvent especially for fats, oils, etc. Resembles chloroform in odour.

Igi, *J. Chem. Ind. Japan*, 1920, **23**, 1217.

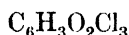
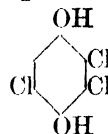
Clayton Aniline Co., D.R.P., 222,622, (*Chem. Zentr.*, 1910, II, 121).

Sastry, *Chem. Zentr.*, 1916, II, 306.**Trichloroguaiacol.**

See under Trichlorocatechol.

**Trichlorohydrin.**

See 1 : 2 : 3-Trichloropropane.

**Trichlorohydroquinone**

MW, 213.5

Prisms from H<sub>2</sub>O. M.p. 136°. Sol. EtOH, Et<sub>2</sub>O. Sol. 160 parts H<sub>2</sub>O at 16°. Sublimes in plates.

Di-Et ether : 2 : 3 : 5-trichloro-1 : 4-diethoxybenzene. C<sub>10</sub>H<sub>11</sub>O<sub>2</sub>Cl<sub>3</sub>. MW, 269.5. Needles from EtOH. M.p. 68.5°.

Diacetyl : sublimes in needles. M.p. 153°.

Conant, Fieser, *J. Am. Chem. Soc.*, 1923, **45**, 2206.

Graebe, *Ann.*, 1868, **146**, 27.Levy, Schultz, *Ann.*, 1881, **210**, 153.**2 : 2 : 2-Trichloro-1-hydroxy-acetylpropylamine.**

See Chloralacetamide.

**2 : 2 : 2-Trichloro-1-hydroxyethylacetamide.**

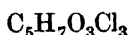
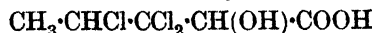
See Chloralacetamide.

**2 : 2 : 2-Trichloro-1-hydroxyethylformamide.**

See Chloralacetamide.

**3 : 3 : 3-Trichloro-2-hydroxypropyl phenyl Ketone.**

See Chloralacetophenone.

**2 : 2 : 3-Trichloro-1-hydroxyvaleric Acid**

MW, 221.5

Tablets. M.p. 140°. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Prac. insol. H<sub>2</sub>O.

Et ester : C<sub>7</sub>H<sub>11</sub>O<sub>3</sub>Cl<sub>3</sub>. MW, 249.5. Prisms. M.p. 40°. B.p. 255° decomp.

**Amide**:  $C_6H_5O_2NCl_3$ . MW, 220.5. Cryst. from  $C_6H_6$  or pet. ether. M.p. 119°. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ ,  $C_6H_6$ .

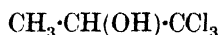
**Nitrile**: butylchloral cyanhydrin.  $C_5H_6ONCl_3$ . MW, 202.5. Plates from aq. HCl. M.p. 101–2°. B.p. 230° decomp. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ ,  $C_6H_6$ . **Acetyl**: yellow oil. B.p. 240–52° decomp.

**Acetyl**: needles +  $1H_2O$ . M.p. 84°. Anhyd. comp. is a syrup.

Pinner, Klein, *Ber.*, 1878, 11, 1488.

Pinner, Bischoff, *Ann.*, 1875, 179, 99.

### 1 : 1 : 1-Trichloroisopropyl Alcohol (Isopral)



$C_3H_5OCl_3$  MW, 163.5

M.p. 50–1°. B.p. 161.8°/773 mm. Sol. ord. org. solvents. Spar. sol.  $H_2O$ . Resembles camphor in odour.

Bayer, D.R.P., 151,545, (*Chem. Zentr.*, 1904, I, 1586).

Garzarolli-Thurnlackh, *Ann.*, 1881, 210, 77.

Serantes, *Anales de la asociacion quimica Argentina*, 1924, 12, 199 (*Bibl.*).

### 2 : 2 : 2-Trichlorolactic Acid



$C_3H_3O_3Cl_3$  MW, 193.5

Prisms. M.p. anhyd. 124°. B.p. 140–70°/45 mm. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ .  $k = 4.65 \times 10^{-3}$  at 25°.

**Me ester**:  $C_4H_5O_3Cl_3$ . MW, 207.5. Liq. B.p. 98–100°/12 mm.

**Et ester**:  $C_5H_7O_3Cl_3$ . MW, 221.5. Plates. M.p. 66–7°. B.p. 233–7°. Insol.  $H_2O$ . Sol. alkalis. **Et ether**:  $C_7H_{11}O_3Cl_3$ . MW, 249.5. Liq. B.p. 128–30°/12 mm.  $D_4^{20}$  1.34115.

**Acetyl**: b.p. 121–121.5°/16 mm.  $D^{20}$  1.367. **Urethane**: needles from  $Et_2O$ –pet. ether. M.p. 57.5°.

**Propyl ester**:  $C_6H_9O_3Cl_3$ . MW, 235.5. B.p. 248–50°, 115–17°/12 mm.  $D_4^{20}$  1.51628.

**Isobutyl ester**:  $C_7H_{11}O_3Cl_3$ . MW, 249.5. B.p. 236–8°, 111–12°/12 mm.  $D_4^{20}$  1.53216.

**Amide**:  $C_3H_4O_2NCl_3$ . MW, 192.5. Needles. M.p. 96°. Sol. EtOH,  $Et_2O$ . Spar. sol. cold  $H_2O$ . **O-Acetyl**: needles. M.p. 94–5°.

**Nitrile**: chloral cyanhydrin.  $C_3H_2ONCl_3$ . MW, 174.5. Tablets from  $CS_2$  or  $H_2O$ . M.p. 61°. B.p. 215–20° slight decomp. Sol.  $H_2O$ , EtOH,  $Et_2O$ . **Acetyl**: cryst. M.p. 31°. B.p. 208°. **Urethane**: tablets from EtOH.Aq. M.p. 115–16°.

**Anilide**: prisms from  $CHCl_3$ . M.p. 164–5° decomp.

**Acetyl**: cryst. from  $C_6H_6$ . M.p. 65°.

Kölln, *Ann.*, 1918, 416, 232.

Anschütz, Haslam, *Ann.*, 1889, 253, 129.

Pinner, *Ber.*, 1884, 17, 1997.

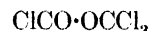
### Trichloromethane.

See Chloroform.

### Trichloromethyl bromide.

See Trichlorobromomethane.

**Trichloromethyl chloroformate** (Diphosgene, perchloromethyl chloroformate, superpalite)



$C_2O_2Cl_4$  MW, 198

Oily liq. B.p. 128°, 49°/50 mm.  $D^{20}$  1.6525.  $n_D^{20}$  1.4566. Decomp. on strong heating to  $COCl_2$ . Very toxic and asphyxiating. Used during Great War as a poison gas under the name of Green Cross.

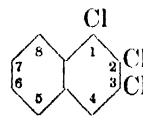
Grignard, Rivat, Urbain, *Compt. rend.*, 1919, 169, 1075, 1143.

Kling, Florentin, Lassieur, Schmutz, *Compt. rend.*, 1919, 169, 1166.

### Trichloromethyl sulphochloride.

See Perchloromethyl Mercaptan.

### 1 : 2 : 3-Trichloronaphthalene



$C_{10}H_5Cl_3$  MW, 231.5

Prisms from  $Et_2O$ –EtOH. M.p. 81°.

Faust, Saame, *Ann.*, 1871, 160, 71.

Armstrong, Wynne, *Chem. News*, 1890, 61, 272.

### 1 : 2 : 4-Trichloronaphthalene.

Needles. M.p. 92°.

Cleve, *Ber.*, 1890, 23, 954.

Armstrong, Wynne, *Chem. News*, 1890, 61, 273.

### 1 : 2 : 5-Trichloronaphthalene.

Needles from EtOH. M.p. 78–78.5°. Sol. EtOH,  $CHCl_3$ , AcOH,  $C_6H_6$ .

Armstrong, Wynne, *Chem. News*, 1889, 59, 188; 1890, 62, 164.

### 1 : 2 : 6-Trichloronaphthalene.

Needles from EtOH. M.p. 92.5°. Sol.  $CHCl_3$ .

Armstrong, Wynne, *Chem. News*, 1889, 59, 189; 1890, 61, 274.

**1 : 2 : 7-Trichloronaphthalene.**

Needles from EtOH. M.p. 88°.

Armstrong, Wynne, *Chem. News*, 1889, 59, 189; 1895, 71, 254.**1 : 2 : 8-Trichloronaphthalene.**

Needles from EtOH. M.p. 83°.

Armstrong, Wynne, *Chem. News*, 1895, 71, 253.**1 : 3 : 5-Trichloronaphthalene.**

Yellow needles from EtOH. M.p. 103° (94°). Sol. common org. solvents.

Friedländer, Karamessinis, Schenk, *Ber.*, 1922, 55, 49.Armstrong, Wynne, *Chem. News*, 1890, 61, 273; 1896, 73, 55.**1 : 3 : 6-Trichloronaphthalene.**

Needles. M.p. 80·5°.

Armstrong, Wynne, *Chem. News*, 1890, 62, 164; 1895, 71, 254.**1 : 3 : 7-Trichloronaphthalene.**

Needles from EtOH. M.p. 113°. Mod. sol. EtOH.

Armstrong, Wynne, *Chem. News*, 1890, 61, 93, 275; 1897, 76, 69.**1 : 3 : 8-Trichloronaphthalene.**

Needles from EtOH. M.p. 89·5°.

Armstrong, Wynne, *Chem. News*, 1890, 61, 94.**1 : 4 : 5-Trichloronaphthalene.**

Needles. M.p. 131°. Sol. AcOH, warm EtOH.

Armstrong, Wynne, *Chem. News*, 1890, 61, 273.I.G., F.P., 683,792, (*Chem. Zentr.*, 1930, II, 1446).**1 : 4 : 6-Trichloronaphthalene.**

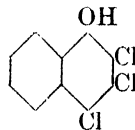
Needles from EtOH. M.p. 65°. Spar. sol. boiling EtOH.

Armstrong, Wynne, *Chem. News*, 1890, 61, 94, 275; 1890, 62, 162.**1 : 6 : 7-Trichloronaphthalene.**

Needles. M.p. 109·5°.

Armstrong, Wynne, *Chem. News*, 1890, 61, 275; 1895, 71, 253.**2 : 3 : 6-Trichloronaphthalene.**

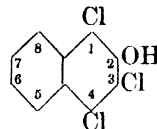
Cryst. from MeOH-EtOH. M.p. 145° (90·5-91°).

Franzen, Stäuble, *J. prakt. Chem.*, 1922, 103, 377.Armstrong, Wynne, *Chem. News*, 1890, 61, 275; 1890, 62, 163.**2 : 3 : 4-Trichloro-1-naphthol** $C_{10}H_5OCl_3$ 

MW, 247·5

Needles from ligroin. M.p. 168° (159-60°). Sol. Et<sub>2</sub>O. Mod. sol. AcOH, warm EtOH.

Acetyl: needles. M.p. 123-4°.

Franzen, Stäuble, *J. prakt. Chem.*, 1922, 103, 385.Zincke, *Ber.*, 1888, 21, 1036.**1 : 3 : 4-Trichloro-2-naphthol** $C_{10}H_5OCl_3$ 

MW, 247·5

Needles. M.p. 162°.

Acetyl: needles from AcOH. M.p. 133·5-134°.

Zincke, *Ber.*, 1888, 21, 3390, 3554.**1 : 4 : 5-Trichloro-2-naphthol.**

Needles from AcOH. M.p. 157-8°.

Acetyl: m.p. 129°.

Armstrong, Rossiter, *Chem. News*, 1891, 63, 136.**2 : 3 : 4-Trichloronitrobenzene** $C_6H_2O_2NCl_3$ 

MW, 226·5

Needles. M.p. 55·5°. Sol. CS<sub>2</sub>. Spar. sol. EtOH.Tiessens, *Rec. trav. chim.*, 1931, 50, 112.Holleman, van Haften, *Rec. trav. chim.*, 1921, 40, 69.**2 : 3 : 5-Trichloronitrobenzene.**

Yellow needles from 80% EtOH. M.p. 45°.

Hodgson, Kershaw, *J. Chem. Soc.*, 1929, 2920.Holleman, van Haften, *Rec. trav. chim.*, 1921, 40, 72.

**2 : 3 : 6-Trichloronitrobenzene.**

Needles from EtOH. M.p. 88–9°. Sol. EtOH. Spar. sol. ligroin.

Beilstein, Kurbatow, *Ann.*, 1878, **192**, 232.  
Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 69.

**2 : 4 : 5-Trichloronitrobenzene.**

Prisms from EtOH or CS<sub>2</sub>. M.p. 57°. B.p. 288°. D<sub>25</sub><sup>20</sup> 1.790. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Spar. sol. EtOH.

Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 71.

Cohen, Bennett, *J. Chem. Soc.*, 1905, **87**, 321.

**2 : 4 : 6-Trichloronitrobenzene.**

Needles from EtOH. M.p. 69°. Spar. sol. EtOH.

Holleman, van Haeften, *Rec. trav. chim.*, 1921, **40**, 74.

**3 : 4 : 5-Trichloronitrobenzene.**

Pale yellow cryst. M.p. 72.5°. Spar. volatile in steam.

Holleman, *Rec. trav. chim.*, 1921, **40**, 69.

**Trichloronitromethane.**

See Chloropicrin.

**Trichlorophenetole.**

See under Trichlorophenol.

**2 : 3 : 4-Trichlorophenol**

C<sub>6</sub>H<sub>3</sub>OCl<sub>3</sub> MW, 197.5

Needles from ligroin. M.p. 83.5°.  $k = 2.22 \times 10^{-8}$  at 25°.

*Me ether* : 2 : 3 : 4-trichloroanisole. C<sub>7</sub>H<sub>5</sub>OCl<sub>3</sub>. MW, 211.5. Prisms from EtOH. M.p. 69.5°.

*Benzoyl* : needles from EtOH. M.p. 141°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 743.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 112.

Hodgson, Kershaw, *J. Chem. Soc.*, 1930, 1421.

**2 : 3 : 5-Trichlorophenol.**

Cryst. M.p. 62°. Very hygroscopic.  $k = 4.33 \times 10^{-8}$  at 25°.

*Me ether* : 2 : 3 : 5-trichloroanisole. Needles from EtOH. M.p. 84°.

*Benzoyl* : needles from ligroin. M.p. 103°.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 114.

Hodgson, Kershaw, *J. Chem. Soc.*, 1929, 2921.

**2 : 3 : 6-Trichlorophenol.**

Needles from pet. ether. M.p. 58°.  $k = 7.37 \times 10^{-7}$  at 25°.

*Benzoyl* : cryst. from EtOH. M.p. 90°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 742.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 113.

**2 : 4 : 5-Trichlorophenol.**

Needles from pet. ether. M.p. 68°. B.p. 244–8°/746 mm.  $k = 3.76 \times 10^{-8}$  at 25°.

*Me ether* : 2 : 4 : 5-trichloroanisole. Needles from EtOH. M.p. 77.5°. B.p. 252–5°/742 mm.

*Benzoyl* : needles from EtOH. M.p. 92–3°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 737.

Kohn, Fink, *Monatsh.*, 1931, **58**, 83.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 114.

**2 : 4 : 6-Trichlorophenol.**

Needles from AcOH. M.p. 69.5°. B.p. 246°. D<sub>25</sub><sup>20</sup> 1.4901.  $k = 3.76 \times 10^{-8}$  at 25°. Sol. EtOH, Et<sub>2</sub>O. Volatile in steam.

*Me ether* : 2 : 4 : 6-trichloroanisole. Needles from EtOH. M.p. 61–2°. B.p. 240°/738 mm. Sublimes.

*Ether* : 2 : 4 : 6-trichlorophenetole. C<sub>8</sub>H<sub>5</sub>OCl<sub>3</sub>. MW, 225.5. Prisms. M.p. 43–4°. B.p. 246°.

*Acetyl* : b.p. 261–2°.

*Propionyl* : b.p. 262.5–264.5°.

*Butyryl* : b.p. 272–5°.

*Benzoyl* : needles from EtOH. M.p. 75.5°.

*p-Nitrobenzoyl* : m.p. 105–6°.

Kohn, Fink, *Monatsh.*, 1931, **58**, 88.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 115.

Datta, Mitter, *J. Am. Chem. Soc.*, 1919, **41**, 2032.

**3 : 4 : 5-Trichlorophenol.**

Needles from ligroin. M.p. 101° (91°). B.p. 271–7°/746 mm.  $k = 1.77 \times 10^{-8}$  at 25°.

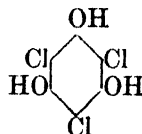
*Me ether* : 3 : 4 : 5-trichloroanisole. M.p. 63°. B.p. 256–61°.

*Benzoyl* : needles from EtOH. M.p. 120°.

Holleman, *Rec. trav. chim.*, 1920, **39**, 740.

Tiessens, *Rec. trav. chim.*, 1931, **50**, 113.

Kohn, Kramer, *Monatsh.*, 1928, **49**, 161.

**Trichlorophloroglucinol**

C<sub>6</sub>H<sub>3</sub>O<sub>3</sub>Cl<sub>3</sub> MW, 229.5

Cryst. from EtOH. M.p. 136°. Sol. EtOH. Prac. insol. H<sub>2</sub>O, cold C<sub>6</sub>H<sub>6</sub>.

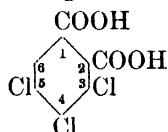
*Di-Me ether* : C<sub>8</sub>H<sub>7</sub>O<sub>3</sub>Cl<sub>3</sub>. MW, 257.5. Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 93–5°. *Acetyl* : m.p. 58–9°.

*Tri-Me ether*:  $C_9H_9O_3Cl_3$ . MW, 271.5. Needles from EtOH. M.p. 130–1°. Volatile in steam.

*Triacetyl*: plates from AcOH.Aq. M.p. 167–8°.

Zincke, Kegel, *Ber.*, 1889, **22**, 1476.

### 3 : 4 : 5-Trichlorophthalic Acid



$C_8H_3O_4Cl_3$  MW, 269.5

Yellow cryst. Heat  $\rightarrow$  anhydride.

*Anhydride*:  $C_8HO_3Cl_3$ . MW, 251.5. M.p. 157°. Sublimes in needles.

Claus, Kautz, *Ber.*, 1885, **18**, 1370.

### 3 : 4 : 6-Trichlorophthalic Acid.

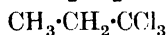
Cryst. from  $H_2O$ . Above 130°  $\rightarrow$  anhydride.

*Anhydride*: m.p. 148°. Sublimes in needles. Hot  $H_2O \rightarrow$  acid.

*Imide*:  $C_8H_2O_2NCl_3$ . MW, 250.5. Needles from EtOH. M.p. 236°. Sol. EtOH,  $Et_2O$ . Mod. sol. hot  $H_2O$ .

Graebe, Rostowzew, *Ber.*, 1901, **34**, 2107.

### 1 : 1 : 1-Trichloropropane



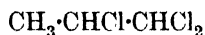
$C_3H_5Cl_3$  MW, 147.5

B.p. 145–50°. Heat with  $Ag_2O + H_2O \rightarrow$  propionic acid.

v. Arkel, *Rec. trav. chim.*, 1932, **51**, 1101.

Spring, Lecrenier, *Bull. soc. chim.*, 1887, **48**, 625.

**1 : 1 : 2-Trichloropropane** ( $\alpha$ -Chloropropylidene chloride)



$C_3H_5Cl_3$  MW, 147.5

Oil. B.p. 140° (132°).  $D^{25}$  1.372.

Klebanski, Wolkenstein, *Chem. Zentr.*, 1935, II, 3298.

Herzfelder, *Ber.*, 1893, **26**, 1258.

**1 : 1 : 3-Trichloropropane** ( $\beta$ -Chloropropylidene chloride)



$C_3H_5Cl_3$  MW, 147.5

B.p. 146–8°.  $D^{15}$  1.362.

Kirrman, Pacaud, Dosque, *Bull. soc. chim.*, 1934, **1**, 864.

Romburgh, *Bull. soc. chim.*, 1882, **37**, 100.

**1 : 2 : 2-Trichloropropane** (Chloroisopropylidene chloride)

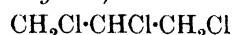


$C_3H_5Cl_3$  MW, 147.5

B.p. 123°.  $D^{25}$  1.318.

Herzfelder, *Ber.*, 1893, **26**, 1259.

**1 : 2 : 3-Trichloropropane** (Trichlorohydrin, glycerol trichlorohydrin)



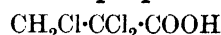
$C_3H_5Cl_3$  MW, 147.5

B.p. 158°.  $D^{15}$  1.417.

Carré, Maucière, *Compt. rend.*, 1931, **192**, 1568.

Fittig, Pfeffer, *Ann.*, 1865, **135**, 359.

### 1 : 1 : 2-Trichloropropionic Acid



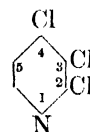
$C_3H_3O_2Cl_3$  MW, 177.5

Prisms from  $CS_2$ . M.p. 50–2°. Sol.  $H_2O$ , EtOH,  $C_6H_6$ .

*Ester*:  $C_5H_7O_2Cl_3$ . MW, 205.5. B.p. 121°/55 mm.  $D^{25}$  1.36.  $n_D^{25}$  1.458.

Berlande, *Bull. soc. chim.*, 1925, **37**, 1385.

### 2 : 3 : 4-Trichloropyridine



$C_5H_2NCl_3$  MW, 182.5

Needles. M.p. 45–7°. Sublimes in vacuo.

Graf, *J. prakt. Chem.*, 1933, **138**, 235.

### 2 : 3 : 5-Trichloropyridine.

Needles from 50% EtOH. M.p. 50°. Sol.  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. EtOH, pet. ether. Insol.  $H_2O$ , dil. acids.

Räth, *Ann.*, 1931, **486**, 78.

Fischer, Chur, *J. prakt. Chem.*, 1916, **93**, 371.

### 2 : 4 : 6-Trichloropyridine.

M.p. 33°.

Graf, *J. prakt. Chem.*, 1932, **133**, 44.

### 3 : 4 : 5-Trichloropyridine.

Needles from EtOH.Aq. M.p. 76–7°. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , pet. ether. Mod. sol. EtOH. Spar. sol.  $H_2O$ . Sol. conc. min. acids.

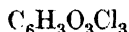
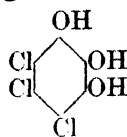
$B_2HgCl_2$ : needles. M.p. 168–70°.

Dohrn, Diedrich, *Ann.*, 1932, **494**, 298.

Sell, *J. Chem. Soc.*, 1905, **87**, 800.



## Trichloropyrogallol



MW, 229.5

Needles +  $3H_2O$ . M.p. anhyd.  $185^\circ$  ( $177^\circ$ ). Sol. EtOH, Et<sub>2</sub>O, hot H<sub>2</sub>O. Spar.  $CHCl_3$ ,  $CCl_4$ ,  $CS_2$ ,  $C_6H_6$ .

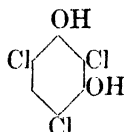
*Tri-Me ether*:  $C_9H_9O_3Cl_3$ . MW, 271.5. Cryst. from EtOH. M.p.  $54^\circ$ . Spar. volatile in steam.

*Triacetyl*: needles from AcOH.Aq. M.p.  $122^\circ$ .

Hantzsch, Schniter, *Ber.*, 1887, **20**, 2036.

Webster, *J. Chem. Soc.*, 1884, **45**, 205.

## 2 : 4 : 6-Trichlororesorcinol



MW, 213.5

Cryst. from H<sub>2</sub>O. M.p.  $83^\circ$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

*Diacetyl*: prisms from EtOH. M.p.  $116^\circ$ .

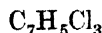
Likhoshesterov, *Chem. Abstracts*, 1934, **28**, 1675.

Zincke, Rabinowitsch, *Ber.*, 1890, **23**, 3767.

 $\omega$ -Trichlorotoluene.

See Benzotrichloride.

## 2 : 3 : 4-Trichlorotoluene



MW, 195.5

Needles from EtOH. M.p.  $41^\circ$ . B.p.  $231-2^\circ/716$  mm. Sol. common org. solvents. Dil.  $HNO_3 \rightarrow$  2 : 3 : 4-trichlorobenzoic acid.

Seelig, *Ann.*, 1887, **237**, 132, 156.

## 2 : 3 : 5-Trichlorotoluene.

Needles from EtOH. M.p.  $45-6^\circ$ . B.p.  $229-31^\circ/757$  mm. Dil.  $HNO_3 \rightarrow$  2 : 3 : 5-trichlorobenzoic acid.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1343.

## 2 : 3 : 6-Trichlorotoluene.

Needles from EtOH. M.p.  $45-6^\circ$ . Dil.  $HNO_3 \rightarrow$  2 : 3 : 6-trichlorobenzoic acid.

See previous reference.

## 2 : 4 : 5-Trichlorotoluene.

Needles from EtOH. M.p.  $82^\circ$ . B.p.  $229-30^\circ/716$  mm.,  $152^\circ/25$  mm. Dil.  $HNO_3 \rightarrow$  2 : 4 : 5-trichlorobenzoic acid. Sublimes in needles.

Fichter, Glanztein, *Ber.*, 1916, **49**, 2481.

Feldman, Kopeliowitsch, *Chem. Zentr.*, 1936, **I**, 2550.

Qvist, Holmberg, *Chem. Zentr.*, 1932, **II**, 2816.

B.D.C., E.P., 169,025, (*Chem. Zentr.*, 1922, **IV**, 376).

## 2 : 4 : 6-Trichlorotoluene.

Needles from EtOH. M.p.  $38^\circ$  ( $33-4^\circ$ ). Volatile in steam. Dil.  $HNO_3 \rightarrow$  2 : 4 : 6-trichlorobenzoic acid.

Bureš, Trpišovska, *Chem. Zentr.*, 1936, **I**, 1209.

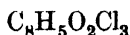
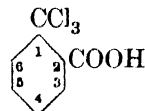
Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1335.

## 3 : 4 : 5-Trichlorotoluene.

M.p.  $44.5-45.5^\circ$ . B.p.  $245.5-247^\circ$ . Volatile in steam. Dil.  $HNO_3 \rightarrow$  3 : 4 : 5-trichlorobenzoic acid.

Cohen, Dakin, *J. Chem. Soc.*, 1902, **81**, 1337.

$\omega$ -Trichloro-*o*-toluic Acid (Benzotrichloride-*o*-carboxylic acid)



MW, 239.5

Needles from  $C_6H_6$ . M.p.  $141-4^\circ$ .

*Me ester*:  $C_9H_7O_2Cl_3$ . MW, 253.5. B.p.  $125^\circ/1$  mm.

*Et ester*:  $C_{10}H_9O_2Cl_3$ . MW, 267.5. Mobile liq. Decomp. on dist.

*Chloride*:  $C_8H_4OCl_4$ . MW, 258. Cryst. from pet. ether. M.p.  $87^\circ$ . B.p.  $145-55^\circ/20$  mm.

*Nitrile*: *o*-cyanobenzotrichloride.  $C_8H_4NCl_3$ . MW, 220.5. Cryst. from EtOH. M.p.  $94-5^\circ$ . B.p.  $280^\circ$ . Conc. HCl in sealed tube  $\rightarrow$  phthalic acid.

*Anilide*: cryst. from  $C_6H_6$ . M.p.  $165-70^\circ$ .

Davies, Perkin, *J. Chem. Soc.*, 1922, **121**, 2213.

Ott, *Ber.*, 1922, **55**, 2123.

$\omega$ -Trichloro-*m*-toluic Acid (Benzotrichloride-*m*-carboxylic acid).

Plates. M.p.  $142^\circ$ . Sol.  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. formic acid.

*Me ester* : needles from  $\text{Me}_2\text{CO-MeOH}$ . M.p. 55°.

*Chloride* : oil. B.p. 287°/754 mm.

See first reference above.

**ω-Trichloro-*p*-toluic Acid** (*Benzotrichloride-p-carboxylic acid*).

Plates. M.p. 196-7°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. formic acid.

*Me ester* : plates. M.p. 55°.

*Et ester* : plates. M.p. 57°.

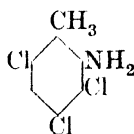
*Chloride* : oil. B.p. 296°/756 mm.

*Amide* :  $\text{C}_6\text{H}_6\text{ONCl}_3$ . MW, 238.5. Needles from  $\text{C}_6\text{H}_6$ . M.p. 180°.

Davies, Perkin, *J. Chem. Soc.*, 1922, 121, 2214.

Böeseke, Gelissen, *Rec. trav. chim.*, 1924, 43, 869.

**3 : 4 : 6-Trichloro-*o*-toluidine**



$\text{C}_7\text{H}_6\text{NCl}_3$  MW, 210.5

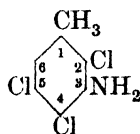
Needles from EtOH. M.p. 89°. Volatile in steam.

*N-Acetyl* : 3 : 4 : 6-trichloroaceto-*o*-toluidide. Needles from EtOH.Aq. M.p. 199°.

*N-Benzoyl* : plates from EtOH. M.p. 230°.

Levy, Stephen, *J. Chem. Soc.*, 1931, 78.

**2 : 4 : 5-Trichloro-*m*-toluidine**



$\text{C}_7\text{H}_6\text{NCl}_3$  MW, 210.5

Needles from EtOH. M.p. 94-5°.

*N-Acetyl* : 2 : 4 : 5-trichloroacet-*m*-toluidide. Needles from EtOH. M.p. 190-1°.

*N-Benzoyl* : needles. M.p. 213°. Spar. sol. hot EtOH.

Seelig, *Ann.*, 1887, 237, 141.

Schultz, *Ann.*, 1877, 187, 278.

**2 : 4 : 6-Trichloro-*m*-toluidine.**

Needles from EtOH. M.p. 85° (77-8°).

*N-Acetyl* : 2 : 4 : 6-trichloroacet-*m*-toluidide. Needles from EtOH. M.p. 192° (181°).

*N-Diacetyl* : m.p. 81-2°.

*N-Benzoyl* : leaflets. M.p. 218°.

Bureš, Trpišovska, *Chem. Zentr.*, 1936, I, 1209.

Cohen, Dakin, *J. Chem. Soc.*, 1902, 81, 1335.

**2 : 5 : 6-Trichloro-*m*-toluidine.**

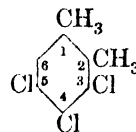
Cryst. from MeOH.Aq. M.p. 66-7°.

Cohen, Dakin, *J. Chem. Soc.*, 1904, 85, 1281.

**3 : 4 : 5-Trichloroveratrol.**

See under 3 : 4 : 5-Trichlorocatechol.

**3 : 4 : 5-Trichloro-*o*-xylene**



$\text{C}_8\text{H}_7\text{Cl}_3$  MW, 209.5

Needles from EtOH. M.p. 96°. B.p. 261°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , pet. ether, hot EtOH. Volatile in steam.

Hinkel, *J. Chem. Soc.*, 1920, 117, 1301.

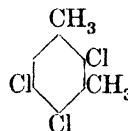
Cf. Claus, Kautz, *Ber.*, 1885, 18, 1369.

**3 : 5 : 6-Trichloro-*o*-xylene.**

Cryst. from EtOH. M.p. 47-5°. B.p. 230-40°. Sol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , AcOEt,  $\text{C}_6\text{H}_6$ , pet. ether. Mod. sol. MeOH, EtOH.

Hinkel, *J. Chem. Soc.*, 1920, 117, 1300.

**2 : 4 : 5-Trichloro-*m*-xylene**



$\text{C}_8\text{H}_7\text{Cl}_3$  MW, 209.5

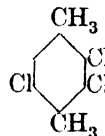
M.p. 95-6°. B.p. 225-60°.

I.G., F.P., 650,732, (*Chem. Abstracts*, 1929, 23, 3233).

General Aniline Works Inc., U.S.P., 1,796,108, (*Chem. Zentr.*, 1931, I, 3610).

Cf. Bureš, Borgmann, *Chem. Zentr.*, 1928, I, 1171.

**2 : 3 : 5-Trichloro-*p*-xylene**

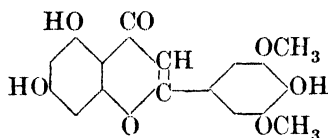


$\text{C}_8\text{H}_7\text{Cl}_3$  MW, 209.5

Needles. M.p. 96°. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether. Insol.  $\text{H}_2\text{O}$ .

Bureš, Rubeš, *Chem. Zentr.*, 1929, I, 507.

**Tricin** (*Tricetin 3' : 5'-dimethyl ether, 5 : 7 : 4'-trihydroxy-3' : 5'-dimethoxyflavone*)



$C_{17}H_{14}O_7$  MW, 330

Colouring matter of leaves of Khapli wheat. Yellow needles from AcOH.Aq. M.p. 291–2°. Conc.  $H_2SO_4 \rightarrow$  yellow sol. Alc.  $FeCl_3 \rightarrow$  reddish-brown col.

4'-Benzyl ether:  $C_{24}H_{20}O_7$ . MW, 420. Pale orange prismatic needles from  $Me_2CO$ .Aq. M.p. 234°. Conc.  $H_2SO_4 \rightarrow$  pale yellow col. Alc.  $FeCl_3 \rightarrow$  greenish-brown col.

Diacetyl deriv.: pale yellow needles. M.p. 211–13°.

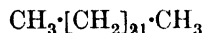
Triacetyl: needles from EtOH–AcOH. M.p. 251–4°.

Tri-Me ether: 5 : 7 : 3' : 4' : 5'-pentamethoxyflavone.  $C_{20}H_{20}O_7$ . MW, 372. Needles from MeOH. M.p. 192–3°.

Anderson, *Chem. Zentr.*, 1932, II, 3899; 1933, II, 2012.

Gulati, Venkataraman, *J. Chem. Soc.*, 1933, 1644.

### Tricosane

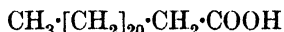


$C_{23}H_{48}$  MW, 324

Present in Pennsylvania petroleum. Leaflets from EtOH. M.p. 48°. B.p. 234°/15 mm.

Read, Andrews, *J. Soc. Chem. Ind.*, 1920, 39, 290T.

### Tricosanic Acid



$C_{23}H_{46}O_2$  MW, 354

Cryst. from  $C_6H_6$ . M.p. 78–9° (80–1°).

Me ester:  $C_{24}H_{48}O_2$ . MW, 368. Cryst. from  $Me_2CO$ . M.p. 55–6°.

Et ester:  $C_{25}H_{50}O_2$ . MW, 382. Cryst. from  $Me_2CO$ . M.p. 52–3°. B.p. 198–9°/0.27 mm.

Nitrile:  $C_{23}H_{45}N$ . MW, 335. Cryst. from  $Me_2CO$ . M.p. 53.5–54.5°.

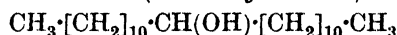
Ashton, Robinson, Smith, *J. Chem. Soc.*, 1936, 285.

Levene, Taylor, *J. Biol. Chem.*, 1924, 59, 905.

### Tricosanol-1.

See Tricosyl Alcohol.

### Tricosanol-12 (Diundecylcarbinol)

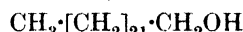


$C_{23}H_{48}O$  MW, 340

Cryst. M.p. 75.5°. Sol. AcOH, pet. ether.

Grün, Ulbrich, Krezil, *Z. angew. Chem.*, 1926, 39, 424.

### Tricosyl Alcohol (Tricosanol-1)



$C_{23}H_{48}O$  MW, 340

Cryst. from  $Me_2CO$ . M.p. 73.5–74.5°. B.p. 191–3°/0.7 mm.

Levene, Taylor, *J. Biol. Chem.*, 1924, 59, 915.

### Tricyanomethane.

See Cyanoform.

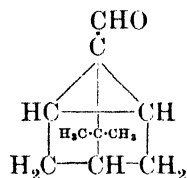
### 1 : 2 : 3-Tricyanopropane.

See under Tricarballic Acid.

### Tricyanotrimethylamine.

See under Triglycolamidic Acid.

### Tricyclal



$C_{10}H_{14}O$  MW, 150

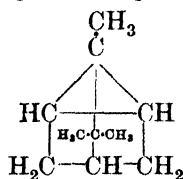
M.p. 85–90°. B.p. 113–15°/31 mm.

Semicarbazone: needles from EtOH.Aq. M.p. 219–20° decomp.

Azine: prisms from EtOH. M.p. 171–2°.

Lipp, *Ber.*, 1920, 53, 778.

**Tricyclene** (1 : 2 : 2-Trimethyl-3 : 6-methylenebicyclo-[0, 1, 3]-hexane, cyclene)



$C_{10}H_{16}$  MW, 136

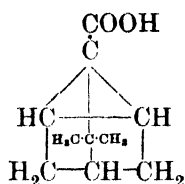
Cryst. from EtOH. M.p. 64–5° (67–8°). B.p. 152–3°.  $D^{20}_D$  0.8268.  $n^{20}_D$  1.4296.

Nametkin, Zabrodin, *Ann.*, 1925, 441, 185.

Lipp, *Ber.*, 1920, 53, 779.

Komppa, *Ber.*, 1929, 62, 1369.

Chem. Fabrik a. Actien, D.R.P., 353,933, (*Chem. Zentr.*, 1922, IV, 499).

**Tricyclic Acid** (*Dehydrocamphenilic acid*) $\text{C}_{10}\text{H}_{14}\text{O}_2$ 

MW, 166

Plates from EtOH or  $\text{C}_6\text{H}_6$ . M.p. 148–9°. B.p. 262–4°, 145°/12 mm. Sol. EtOH,  $\text{C}_6\text{H}_6$ , pet. ether. Insol.  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_{11}\text{H}_{16}\text{O}_2$ . MW, 180. M.p. 45·5° (38°). B.p. 99°/14 mm.  $D_4^{20}$  1·0255.  $n_D^{42.6}$  1·46953.

*Et ester*:  $\text{C}_{12}\text{H}_{18}\text{O}_2$ . MW, 194. B.p. 100–1°/10 mm.  $D_4^{20}$  1·0143.  $n_D^{20}$  1·47299.

*Chloride*:  $\text{C}_{10}\text{H}_{13}\text{OCl}$ . MW, 184·5. M.p. 37·5–38·5°. B.p. 116–17°/15 mm.

*Amide*:  $\text{C}_{10}\text{H}_{15}\text{ON}$ . MW, 165. Leaflets from  $\text{C}_6\text{H}_6$ . M.p. 117–18°.

*Nitrile*:  $\text{C}_{10}\text{H}_{13}\text{N}$ . MW, 147. M.p. 65–70°. B.p. 100–2°/12 mm.

Lipp, *Ber.*, 1920, 53, 774.

Komppa, *Ber.*, 1929, 62, 1366.

**Tridecanal.**

See Tridecyl Aldehyde.

**Tridecane** $\text{C}_{13}\text{H}_{28}$ 

MW, 184

M.p. – 6·2°. B.p. 234°, 130°/30 mm., 114°/15 mm.  $D_4^{20}$  0·7571.

Krafft, *Ber.*, 1882, 15, 1699.

**Tridecanol-1.**

See Tridecyl Alcohol.

**Tridecanol-2.**

See Methylundecylcarbinol.

**Tridecanol-3.**

See Ethyl-*n*-decylcarbinol.

**Tridecanone-2.**

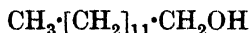
See Methyl undecyl Ketone.

**Tridecanone-3.**

See Ethyl *n*-decyl Ketone.

**2-Tridecylacrylic Acid.**

See Gaidic Acid.

**Tridecyl Alcohol** (*Tridecanol-1*) $\text{C}_{13}\text{H}_{28}\text{O}$ 

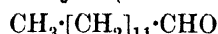
MW, 200

M.p. 30·5°. B.p. 155–6°/15 mm., 117°/0·5 mm.

*Propionyl*: tridecyl propionate. M.p. – 0·4°. B.p. 195°/30 mm.  $D_4^{25}$  0·8574.  $n_D^{20}$  1·4363.

4'-Iodoxenylurethane: m.p. 144–144·5°.

Levene, West, Scheer, *J. Biol. Chem.*, 1915, 20, 528.

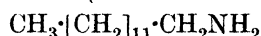
**Tridecyl Aldehyde** (*Tridecanal*) $\text{C}_{13}\text{H}_{26}\text{O}$ 

MW, 198

M.p. 14°. B.p. 156°/23 mm.

*Oxime*: needles from EtOH.Aq. M.p. 80·5°. *Semicarbazone*: plates from EtOH. M.p. 106°.

Le Sueur, *J. Chem. Soc.*, 1905, 87, 1903.

**Tridecylamine** (*1-Aminotridecane*) $\text{C}_{13}\text{H}_{29}\text{N}$ 

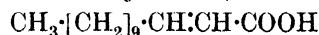
MW, 199

M.p. 27°. B.p. 265°. Sol. EtOH,  $\text{Et}_2\text{O}$ .

Lutz, *Ber.*, 1886, 19, 1436.

Blau, *Monatsh.*, 1905, 26, 101.

**1-Tridecyclic Acid** (*2-*n*-Decylacrylic acid, 1-dodecylene-1-carboxylic acid*)

 $\text{C}_{13}\text{H}_{24}\text{O}_2$ 

MW, 212

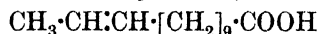
B.p. 167–71°/2 mm.  $D_4^{20}$  0·8995.  $n_D^{20}$  1·46121.

*Chloride*:  $\text{C}_{13}\text{H}_{23}\text{OCl}$ . MW, 230·5. B.p. 131°/3 mm.  $D_4^{20}$  0·9380.

*Amide*:  $\text{C}_{13}\text{H}_{25}\text{ON}$ . MW, 211. M.p. 116°.

Zaar, *Chem. Abstracts*, 1930, 24, 2108.

**10-Tridecyclic Acid** (*Isotridecyclic acid, 2-dodecylene-12-carboxylic acid*)

 $\text{C}_{13}\text{H}_{24}\text{O}_2$ 

MW, 212

Plates from pet. ether. M.p. 28–9°. B.p. 183–5°/13·5 mm., 161–2°/4 mm.

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, 10, 122.

**11-Tridecyclic Acid** (*1-Dodecylene-12-carboxylic acid*)

 $\text{C}_{13}\text{H}_{24}\text{O}_2$ 

MW, 212

Plates from EtOH.Aq. M.p. 38–9°. B.p. 192°/20 mm., 185°/15 mm., 162°/3 mm. Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , pet. ether.

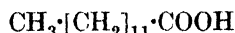
*Me ester*:  $\text{C}_{14}\text{H}_{26}\text{O}_2$ . MW, 226. B.p. 143°/8 mm., 133°/3 mm.  $D_4^{20}$  0·8819.  $n_D^{20}$  1·4438.

*Et ester*:  $\text{C}_{15}\text{H}_{28}\text{O}_2$ . MW, 240. B.p. 150°/8 mm.  $D_4^{15}$  0·880.

Chuit, Boelsing, Hausser, Malet, *Helv. Chim. Acta*, 1927, 10, 118.

Tomecko, Adams, *J. Am. Chem. Soc.*, 1927, 49, 529.

**Tridecylic Acid** (*Tridecoic acid, dodecanoic-1-carboxylic acid*)



$\text{C}_{13}\text{H}_{26}\text{O}_2$  MW, 214

Cryst. from  $\text{Me}_2\text{CO}$ . M.p. 44.5–45.5°. B.p. 199–200°/24 mm.

*Zn salt*: needles from isoamyl alcohol. M.p. 128°.

*Et ester*:  $\text{C}_{15}\text{H}_{30}\text{O}_2$ . MW, 242. B.p. 197–8°/60 mm., 178–80°/20 mm., 163–5°/5 mm.

*Propyl ester*:  $\text{C}_{16}\text{H}_{32}\text{O}_2$ . MW, 256. B.p. 194°/30 mm.  $D_4^{25}$  0.8555.  $n_D^{20}$  1.4357.

*Amide*:  $\text{C}_{13}\text{H}_{27}\text{ON}$ . MW, 213. M.p. 100°.

*Nitrile*:  $\text{C}_{13}\text{H}_{25}\text{N}$ . MW, 195. B.p. 275°.

Ruhoff, *Organic Syntheses*, 1936, XVI, 35.

Levene, West, Allen, Scheer, *J. Biol. Chem.*, 1915, 23, 73.

### Triethanolamine.

See 2 : 2' : 2''-Trihydroxytriethylamine.

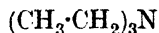
$\omega$  : 2 : 4-Triethoxyacetophenone.

See under Fisetol.

3 : 4 : 5-Triethoxybenzoic Acid.

See under Gallic Acid.

### Triethylamine



$\text{C}_6\text{H}_{15}\text{N}$  MW, 101

M.p. — 114.75°. B.p. 89.4°.  $D_4^{25}$  0.7495,  $D_4^{20}$  0.7255.  $n_D^{20}$  1.4003. Readily oxidised by  $\text{KMnO}_4$ .

*B.HCl*: cryst. from EtOH. M.p. 253–4°.

*B.HBr*: cryst. from  $\text{CHCl}_3$  or EtOH. M.p. 248°.

*B.HI*: m.p. 181°. Sol.  $\text{CHCl}_3$ , EtOH.

*B.HNO\_3*: m.p. 99–100°.

*B.HBr.HgBr\_2*: m.p. 124–5°.

*B\_2,3CHI\_3*: yellow cryst. from EtOH. M.p. 81–3°.

*B,H\_2S*: needles. M.p. 55–7° (closed tube).

*Acetate*: b.p. 162° decomp.

*Picrate*: needles from EtOH. M.p. 172–3°.

Skita, Keil, *Monatsh.*, 1929, 53 and 54, 757.

I.G., E.P., 283,163, (*Chem. Zentr.*, 1929, I, 1509); F.P., 685,345, (*Chem. Zentr.*, 1931, I, 1824).

Hofmann, *Ann.*, 1850, 73, 91.

Rakshit, *J. Am. Chem. Soc.*, 1913, 35, 1782.

### Triethylarsine (Arsenic triethyl)



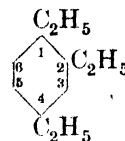
$\text{C}_6\text{H}_{15}\text{As}$  MW, 162

B.p. 140°/736 mm.  $D_4^{20}$  1.150.  $n_D^{20}$  1.467. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Fumes in air and inflames when heated.

Cahours, *Ann.*, 1862, 122, 202.

Hofmann, *Ann.*, 1857, 103, 357.

### 1 : 2 : 4-Triethylbenzene



$\text{C}_{12}\text{H}_{18}$  MW, 162

B.p. 217–18°/755 mm. 99°/15 mm.  $D_4^{20}$  0.8819.  $n_D^{17}$  1.4983.

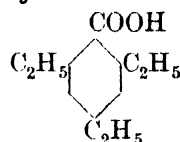
Klages, *J. prakt. Chem.*, 1902, 65, 398.

### 1 : 3 : 5-Triethylbenzene.

B.p. 218°, 95°/14 mm.  $D_4^{20}$  0.8633.  $n_D^{17}$  1.4951.  $\text{CrO}_3 \rightarrow$  trimesic acid.

Gattermann, Fritz, Beck, *Ber.*, 1899, 32, 1122.

### 2 : 4 : 6-Triethylbenzoic Acid



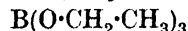
$\text{C}_{13}\text{H}_{18}\text{O}_2$  MW, 206

Plates from ligroin. M.p. 113°.

*Amide*:  $\text{C}_{13}\text{H}_{19}\text{ON}$ . MW, 205. Needles from ligroin. M.p. 155–6°.

Gattermann, Fritz, Beck, *Ber.*, 1899, 32, 1123.

### Triethyl borate (Ethyl borate)

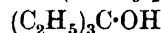


$\text{C}_6\text{H}_{15}\text{O}_3\text{B}$  MW, 146

Liq. B.p. 118.6°.  $D^{26}$  0.864.  $n_D$  1.3808. Rapidly hyd. by H<sub>2</sub>O.

Khotinskii, Pupko, *Chem. Zentr.*, 1929, II, 2763.

### Triethylcarbinol (3-Ethylpentanol-3)



$\text{C}_7\text{H}_{16}\text{O}$  MW, 116

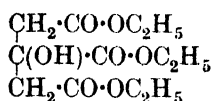
Liq. with penetrating camphor-like odour. B.p. 140–2°.  $D_4^{24}$  0.8407.  $n_D^{23}$  1.4266.

*Allophanate*: m.p. 182–3°.

Moyer, Marvel, *Organic Syntheses*, 1931, XI, 98.

Böeseken, Wildschut, *Rec. trav. chim.*, 1932, 51, 168.

Edgar, Calingaert, Marker, *J. Am. Chem. Soc.*, 1929, 51, 1486.

**Triethyl citrate**

$\text{C}_{12}\text{H}_{20}\text{O}_7$  MW, 276

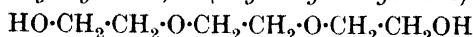
Oil. B.p.  $294^\circ$ ,  $230^\circ/100$  mm.,  $212^\circ/30-5$  mm.  $D_4^{20}$  1.1369.  $n_D^{20}$  1.44554.

*Et ether*:  $\text{C}_{14}\text{H}_{24}\text{O}_7$ . MW, 304. Oil. B.p.  $237-8^\circ/150$  mm.  $D_4^{20}$  1.1022.  $n_D^{20}$  1.4484.

*Acetyl*: 2-acetoxytricarballic triethyl ester. B.p.  $197^\circ/15$  mm. Sol. EtOH, Et<sub>2</sub>O.

Conen, *Ber.*, 1879, 12, 1653.

**Triethylene Glycol** (*Ethylene glycol di-β-hydroxyethyl ether, di-β-hydroxyethoxyethane*)



$\text{C}_6\text{H}_{14}\text{O}_4$  MW, 150

Liq. B.p.  $285^\circ$ ,  $165^\circ/14$  mm.,  $134^\circ/2$  mm.  $D_4^{15}$  1.1274.  $n_D^{15}$  1.4578. Misc. with H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O.

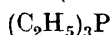
Matignon, Moureau, Dodé, *Bull. soc. chim.*, 1934, 1, 1311.

**Triethylmethane.**

See 3-Ethylpentane.

**Triethyl phosphate.**

See under Phosphoric Acid.

**Triethylphosphine** (*Phosphorus triethyl*)

$\text{C}_6\text{H}_{15}\text{P}$  MW, 118

Colourless liq. with odour of hyacinths. B.p.  $127^\circ$ .  $D_4^{15}$  0.800.  $n_D^{15}$  1.458. Misc. with EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

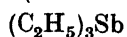
*Add. comp. with CS<sub>2</sub>*: red cryst. from Et<sub>2</sub>O or EtOH. M.p.  $121-2^\circ$  decomp.

*B<sub>2</sub>CuI*: plates from ligroin. M.p.  $39^\circ$ .

*B<sub>2</sub>AuCl*: needles or prisms. M.p.  $80^\circ$ .

*B<sub>2</sub>PdCl<sub>2</sub>*: yellow prisms from Et<sub>2</sub>O.

Slotta, Tschesche, *Ber.*, 1927, 60, 298.

**Triethylstibine** (*Antimony triethyl*)

$\text{C}_6\text{H}_{15}\text{Sb}$  MW, 207

Liq. B.p.  $158.5^\circ/730$  mm.  $D^{16}$  1.3244. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O. Spontaneously inflammable.

Paneth, Loleit, *J. Chem. Soc.*, 1935, 371.

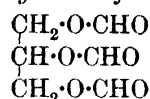
Dyke, Davies, Jones, *J. Chem. Soc.*, 1930, 465.

**Triethylurea.**

See under Urea.

**Trifluoromethane.**

See Fluoroform.

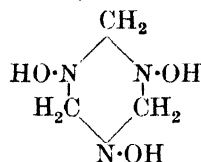
**Triformin** (*Glycerol triformate*)

$\text{C}_6\text{H}_8\text{O}_6$  MW, 176

M.p.  $18^\circ$ . B.p.  $266^\circ/762$  mm.  $D^{18}$  1.320.  $n_D^{18}$  1.4412. Insol. cold H<sub>2</sub>O. Hyd. by hot H<sub>2</sub>O.

Romburgh, *Z. physik. Chem.*, 1910, 70, 459.

**Triformoxime** (*N-Trihydroxytrimethylene-triamine, trimolecular formaldoxime*)



$\text{C}_3\text{H}_9\text{O}_3\text{N}_3$  MW, 135

Amorphous. Insol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Sol. dil. acids, conc. aq. NaOH. Forms coloured salts with Fe, Ni, Mn. H<sub>2</sub>O at  $130^\circ \rightarrow$  formaldoxime.

Scholl, *Ber.*, 1891, 24, 575.

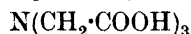
**Trigenic Acid.**

See Trigoniac Acid.

**Trigenolline.**

See Trigonelline.

**Triglycolamidic Acid** (*Trimethylamine-1:1':1''-tricarboxylic acid*)



$\text{C}_6\text{H}_9\text{O}_6\text{N}$  MW, 191

Prisms from H<sub>2</sub>O. Decomp. at  $242^\circ$ . Spar. sol. H<sub>2</sub>O.

*Tri-Me ester*:  $\text{C}_9\text{H}_{15}\text{O}_6\text{N}$ . MW, 233. B.p.  $167^\circ/13$  mm.  $D_4^{17}$  1.2130.  $n_D^{20}$  1.4500.

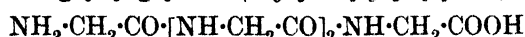
*Tri-Et ester*:  $\text{C}_{12}\text{H}_{21}\text{O}_6\text{N}$ . MW, 275. Viscous oil. B.p.  $193^\circ/18$  mm.

*Triamide*:  $\text{C}_6\text{H}_{12}\text{O}_3\text{N}_4$ . MW, 188. Plates from Alc. NH<sub>3</sub>. Decomp. at  $205-6^\circ$ .

*Trinitrile*: tricyanotrimethylamine.  $\text{C}_6\text{H}_6\text{N}_4$ . MW, 134. Needles from EtOH. M.p.  $125-6^\circ$ .

Curtius, *J. prakt. Chem.*, 1917, 96, 232.

Dubsky, Wensink, *Ber.*, 1916, 49, 1041.

**Triglycylglycine** (*Glycyl-diglycyl-glycine*)

$\text{C}_8\text{H}_{14}\text{O}_5\text{N}_4$  MW, 246

Colourless powder. Darkens at  $220-70^\circ$  with evolution of NH<sub>3</sub>. Very spar. sol. cold H<sub>2</sub>O. Sol hot H<sub>2</sub>O.

*Me ester*:  $\text{C}_9\text{H}_{16}\text{O}_5\text{N}_4$ . MW, 260. Needles or

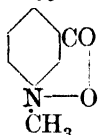
prisms from MeOH. Darkens at 240°. *Hydrochloride*: plates. M.p. 198–200°.

*Et ester*: see Biuret Base.

*Benzoyl*: m.p. 235°.

Fischer, *Ber.*, 1904, **37**, 2501.

**Trigonelline** (N-Methylnicotinic acid betaine, *trigenolline*, *coffeairin*, *gyresin*)



$C_7H_7O_2N$

MW, 137

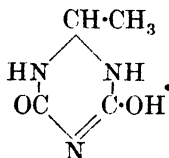
Constituent of fruit and seeds of species of *Strophanthus*, *Cannabis sativa*, coffee, etc. Prisms +  $H_2O$  from EtOH.Aq. M.p. anhyd. 218° decomp. Very sol.  $H_2O$ . Sol. EtOH. Insol.  $Et_2O$ ,  $CHCl_3$ .  $FeCl_3 \rightarrow$  red col.

*B.HCl*: needles or prisms from EtOH. M.p. 245–50° decomp.

*B.HI*: m.p. 220° decomp.

Schulze, *Z. physiol. Chem.*, 1909, **60**, 155.

**Trigonic Acid** (*Trigenic acid*, *ethylidene biuret*)



$C_4H_7O_2N_3$

MW, 129

Prisms or needles from  $H_2O$ . Insol. EtOH. *N-Diacetyl*: m.p. 171–2°.

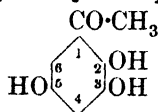
Ostrogovich, Ostrogovich, *Chem. Zentr.*, 1936, II, 476.

Liebig, Wöhler, *Ann.*, 1846, **59**, 296.

**2 : 3 : 4-Trihydroxyacetophenone.**

See Gallacetophenone.

**2 : 3 : 5-Trihydroxyacetophenone**



$C_8H_8O_4$

MW, 168

Yellow needles from AcOH. M.p. 206–7°. Sol. EtOH, hot AcOH. Spar. sol. hot  $C_6H_6$ .

*2 : 3 : 5-Tri-Me ether*: 2 : 3 : 5-trimethoxyacetophenone.  $C_{11}H_{14}O_4$ . MW, 210. Cryst. from ligroin. M.p. 102–3°.

*p-Nitrophenylhydrazone*: cryst. from EtOH.Aq. M.p. 241–2° decomp.

*Triacetyl*: needles from ligroin. M.p. 106–7°.

Mauthner, *J. prakt. Chem.*, 1933, **136**, 214.

**2 : 4 : 5-Trihydroxyacetophenone.**

Red needles from  $H_2O$ . M.p. 200–2° decomp. Sol.  $H_2O$ , hot EtOH,  $Me_2CO$ . Insol.  $C_6H_6$ , petrol.  $FeCl_3 \rightarrow$  green col. Conc.  $H_2SO_4 \rightarrow$  yellowish-green col.  $Ac_2O + AcONa \rightarrow$  6 : 7-diacetoxy-4-methylcoumarin.

*4-Me ether*:  $C_9H_{10}O_4$ . MW, 182. Yellow plates from  $H_2O$ . M.p. 165–6°. *Diacetyl*: needles from  $H_2O$ . M.p. 118–19°.

*5-Me ether*: yellow needles from  $H_2O$ . M.p. 166°.  $FeCl_3 \rightarrow$  red col. *Diacetyl*: needles from  $H_2O$ . M.p. 127–8°.

*4 : 5-Di-Me ether*:  $C_{10}H_{12}O_4$ . MW, 196. Yellow needles from  $H_2O$ . Cryst. from toluene. M.p. 114–15°. *Oxime*: cryst. from EtOH.Aq. M.p. 162°. *Acetyl*: needles from EtOH.Aq. M.p. 147°.

*2 : 4 : 5-Tri-Me ether*: cryst. from  $H_2O$ , EtOH.Aq., or toluene. M.p. 102°. *Semicarbazone*: cryst. from EtOH.Aq. M.p. 206°.

*Diacetyl deriv.*: prisms from  $C_6H_6$ . M.p. 165–6°.

*2 : 4 : 5-Triacetyl*: cryst. from  $CCl_4$ . M.p. 110–11°. *Oxime*: cryst. from EtOH.Aq. M.p. 126–7°. *Semicarbazone*: plates from EtOH. M.p. 186–8°.

Smith, Haller, *J. Am. Chem. Soc.*, 1934, **56**, 237.

Mauthner, *Chem. Abstracts*, 1934, **28**, 3392.

**2 : 4 : 6-Trihydroxyacetophenone.**

See Phloracetophenone.

**3 : 4 : 5-Trihydroxyacetophenone** (*5-Acetyroygallol*).

Needles from  $H_2O$ . M.p. 187–8°. Sol. EtOH,  $Et_2O$ , AcOH,  $Me_2CO$ . Spar. sol.  $C_6H_6$ . Insol. pet. ether, ligroin. Aq.  $FeCl_3 \rightarrow$  green col. Alc.  $FeCl_3 \rightarrow$  blue col.

*3 : 4 : 5-Tri-Me ether*: needles from ligroin. M.p. 72° (78°). *Oxime*: needles from  $H_2O$ . Prisms from EtOH. M.p. 102–3°. *Semicarbazone*: cryst. from  $H_2O$ . M.p. 178–9°.

*3 : 4 : 5-Triacetyl*: needles from ligroin. M.p. 111–12°.

*Semicarbazone*: needles from EtOH. M.p. 216–17°.

*p-Nitrophenylhydrazone*: red cryst. from AcOH.Aq. Decomp. at 260°.

Mauthner, *J. prakt. Chem.*, 1927, **115**, 137.

Bogert, Isham, *J. Am. Chem. Soc.*, 1914, **36**, 523.

**2 : 4 : 6-Trihydroxyanisole.**

See Iretol.

**Trihydroxyanthranol.**

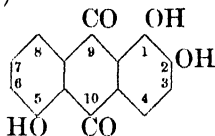
See Tetrahydroxyanthracene.

**1 : 2 : 3-Trihydroxyanthraquinone.**

See Anthragallol.

**1 : 2 : 4-Trihydroxyanthraquinone.**

See Purpurin.

**1 : 2 : 5-Trihydroxyanthraquinone (Hydroxyanthrarufin)** $C_{14}H_8O_5$ 

MW, 256

Red needles from AcOH. M.p. 273–4°. Conc.  $H_2SO_4 \rightarrow$  violet col.2-Me ether:  $C_{15}H_{10}O_5$ . MW, 270. Yellow cryst. from EtOH. M.p. 229°.1:2-Di-Me ether:  $C_{16}H_{12}O_5$ . MW, 284. Orange needles from EtOH. M.p. 230.5–231.5°.1:2:5-Tri-Me ether:  $C_{17}H_{14}O_5$ . MW, 298. Yellow plates from EtOH. M.p. 203–4°.

1:2:5-Triacetyl: yellow needles from EtOH. M.p. 228–9°.

Puntam Becker, Adams, *J. Am. Chem. Soc.*, 1927, **49**, 488.**1 : 2 : 6-Trihydroxyanthraquinone.**

See Flavopurpurin.

**1 : 2 : 7-Trihydroxyanthraquinone.**

See Anthrapurpurin.

**1 : 2 : 8-Trihydroxyanthraquinone (Hydroxychrysazin).**Red needles from AcOH. M.p. 239–40°. Sublimes. Conc.  $H_2SO_4 \rightarrow$  reddish-violet col.2-Me ether:  $C_{15}H_{10}O_5$ . MW, 270. Orange needles from  $CHCl_3$ -MeOH. M.p. 220°.2:8-Di-Me ether:  $C_{16}H_{12}O_5$ . MW, 284. Brownish-yellow cryst. from  $CHCl_3$ -MeOH. M.p. 193°.1:2:8-Tri-Me ether:  $C_{17}H_{14}O_5$ . MW, 298. Yellow needles from MeOH. M.p. 157°.

1:2:8-Triacetyl: yellow needles. M.p. 224°.

Höchst, D.R.P., 196,980, (*Chem. Zentr.*, 1908, I, 1505).Graebe, Thode, *Ann.*, 1906, **349**, 221.**1 : 3 : 8-Trihydroxyanthraquinone.**Yellow prisms. M.p. 275°. Insol. most org. solvents except Py. Sublimes in vacuo at 200°. Conc.  $H_2SO_4 \rightarrow$  yellow col. Red sols. in alkalis.Eder, Hauser, *Helv. Chim. Acta*, 1925, **8**, 134.**1 : 4 : 5-Trihydroxyanthraquinone.**Red needles from  $PhNO_2$  or Py.

4-Acetyl: yellow needles from AcOH. M.p. 165°.

British Celanese, E.P., 346,355, (*Chem. Abstracts*, 1932, **26**, 1948).**1 : 4 : 6-Trihydroxyanthraquinone.**Reddish-brown powder. Does not melt below 300°. Spar. sol. hot  $H_2O$ . Sol. alkalis  $\rightarrow$  violet col.  $Pb(OAc)_4 \rightarrow$  1:4:9:10-diquinone.

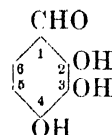
6-Acetyl: cryst. from AcOH.

Crossley, *J. Am. Chem. Soc.*, 1918, **40**, 404.**1 : 3 : 4 - Trihydroxyanthraquinone - 2 - carboxylic Acid.**

See Purpurin-3-carboxylic Acid.

**Trihydroxyanthrone.**

See Tetrahydroxyanthracene.

**2 : 3 : 4-Trihydroxybenzaldehyde** $C_7H_6O_4$ 

MW, 154

Needles from  $H_2O$ . M.p. 161–2°.3:4-Di-Me ether:  $C_9H_{10}O_4$ . MW, 182. Needles from  $H_2O$ , prisms from petrol. M.p. 74°. Phenylhydrazones: yellow prisms from MeOH. M.p. 156°.2:3:4-Tri-Me ether: 2:3:4-trimethoxybenzaldehyde.  $C_{10}H_{12}O_4$ . MW, 196. Prisms from petrol. M.p. 37° (30°). Phenylhydrazones: yellow prisms from MeOH. M.p. 155–6°.2:3:4-Tri-Et ether: 2:3:4-triethoxybenzaldehyde.  $C_{13}H_{18}O_4$ . MW, 238. Cryst. M.p. 70°.Oxime: needles from  $H_2O$ . M.p. 204° decomp.Baker, Smith, *J. Chem. Soc.*, 1931, 2544.Schaaf, Labouchère, *Helv. Chim. Acta*, 1924, **7**, 357.Barger, Ewins, *J. Chem. Soc.*, 1910, **97**, 2258.Gattermann, Berchemann, *Ber.*, 1898, **31**, 1768.**2 : 3 : 5-Trihydroxybenzaldehyde.**2:3-Di-Me ether: cryst. from  $H_2O$ . M.p. 152°.

2:3:5-Tri-Me ether: 2:3:5-trimethoxybenzaldehyde. Cryst. from EtOH.Aq. M.p. 71°.

Smith, Laforge, *J. Am. Chem. Soc.*, 1931, **53**, 3074.**2 : 4 : 5-Trihydroxybenzaldehyde.**Cryst. from  $H_2O$ . M.p. 223°.  $FeCl_3 \rightarrow$  green col.



4-*Me ether* : 2 : 5-dihydroxyanisaldehyde.  $C_8H_8O_4$ . MW, 168. Prisms from EtOH or AcOH. M.p. 209°. Spar. sol. hot EtOH, AcOH,  $Me_2CO$ .  $FeCl_3 \rightarrow$  bluish-green col.

4 : 5-*Di-Me ether* : 6-hydroxyveratric aldehyde.  $C_9H_{10}O_4$ . MW, 182. Needles from  $H_2O$ . Plates from EtOH.Aq. M.p. 105°.  $FeCl_3 \rightarrow$  green col.

2 : 4 : 5-*Tri-Me ether* : see Asarylaldehyde.

4-*Me-5-Et ether* :  $C_{10}H_{12}O_4$ . MW, 196. Prisms from MeOH. M.p. 112–13°. Sol. EtOH,  $Me_2CO$ . Spar. sol.  $H_2O$ .  $FeCl_3 \rightarrow$  green col.

5-*Me-4-Et ether* : prisms from MeOH. M.p. 91°. *Phenylhydrazone* : m.p. 157–8°.

2 : 4-*Di-Me-5-Et ether* :  $C_{11}H_{14}O_4$ . MW, 210. Needles from MeOH. M.p. 110°.

2 : 4 : 5-*Tri-Et ether* : 2 : 4 : 5-triethoxybenzaldehyde.  $C_{13}H_{18}O_4$ . MW, 238. Cryst. from EtOH. M.p. 95°.

4-*Benzoyl* : plates from EtOH.Aq. M.p. 184°.

2 : 4 : 5-*Triacetyl* : prisms from EtOH. M.p. 115°.

Head, Robertson, *J. Chem. Soc.*, 1930, 2436.

Gattermann, Köbner, *Ber.*, 1899, 32, 282.

**2 : 4 : 6-Trihydroxybenzaldehyde** (*Phloroglucinaldehyde*).

Needles +  $2H_2O$  from  $H_2O$ . Darkens on heating.  $FeCl_3 \rightarrow$  red col.

2-*Me ether* : needles from EtOH. M.p. 200–2°.

4 : 6-*Diacetyl* : plates from ligroin. M.p. 107°.

4-*Me ether* : 2-*benzoyl*, plates from EtOH. M.p. 109°.

4 : 6-*Di-Me ether* : needles or plates from MeOH. M.p. 70–1°. 2-*Benzoyl* : needles from AcOEt. M.p. 148°.

2 : 4 : 6-*Tri-Me ether* : 2 : 4 : 6-trimethoxybenzaldehyde. Needles from EtOH. M.p. 118°. *Oxime* : needles from MeOH. M.p. 201–3°.

*Oxime* : cryst. +  $1H_2O$  from  $H_2O$ . Decomp. at 195°.

*Diacetyl deriv.* : needles from EtOH.Aq. M.p. 102–3°.

*Triacetyl* : prisms from EtOH. M.p. 156–7° (151°).

2-*Benzoyl* : prisms from  $CHCl_3$ . M.p. 198–200°.

Malkin, Nierenstein, *J. Am. Chem. Soc.*, 1931, 53, 241.

Robinson *et al.*, *J. Chem. Soc.*, 1930, 804; 1928, 1457; 1927, 1712; 1925, 1184.

Gattermann, Köbner, *Ber.*, 1899, 32, 280.

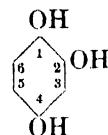
**3 : 4 : 5-Trihydroxybenzaldehyde.**

See Gallaldehyde.

**1 : 2 : 3-Trihydroxybenzene.**

See Pyrogallol.

**1 : 2 : 4-Trihydroxybenzene** (*Hydroxyhydroquinone, hydroxyquinol*)



$C_6H_6O_3$

MW, 126

Plates from  $Et_2O$ . M.p. 140·5°. Sol.  $H_2O$ , EtOH,  $Et_2O$ . Insol.  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Oxidises in air. Conc.  $H_2SO_4 \rightarrow$  green col.  $\rightarrow$  red on warming.  $FeCl_3 + NaOH \rightarrow$  red col.  $Ag_2O \rightarrow$  2-hydroxy-*p*-benzoquinone.

*Picrate* : orange-red needles. M.p. 96°.

1-*Me ether* :  $C_7H_8O_3$ . MW, 140. Prisms from  $C_6H_6$ . M.p. 66–7°. 2 : 4-*Diacetyl* : prisms from MeOH. M.p. 62–4°.

2-*Me ether* : plates from  $H_2O$ . Prisms from  $C_6H_6$ . M.p. 84°. 1 : 4-*Diacetyl* : needles from MeOH. M.p. 93–4°.

1 : 2 : 4-*Tri-Me ether* :  $C_9H_{12}O_3$ . MW, 168. Liq. B.p. 247°.

1 : 2-*Methylene ether* : see Sesamol.

2-*Et ether* :  $C_8H_{10}O_3$ . MW, 154. Prisms from EtOH. M.p. 112·5°.

1 : 2-*Di-Et ether* :  $C_{10}H_{14}O_3$ . MW, 182. Cryst. from  $C_6H_6$  or EtOH. M.p. 65–7°.

1 : 2 : 4-*Tri-Et ether* :  $C_{12}H_{18}O_3$ . MW, 210. Needles from EtOH.Aq. M.p. 34°.

1 : 2 : 4-*Triacetyl* : cryst. from EtOH. M.p. 96–7°.

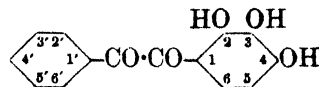
Healey, Robinson, *J. Chem. Soc.*, 1934, 1626.

Vliet, *Organic Syntheses*, Collective Vol. I, 310.

**1 : 3 : 5-Trihydroxybenzene.**

See Phloroglucinol.

**2 : 3 : 4-Trihydroxybenzil**



$C_{14}H_{10}O_5$

MW, 258

Needles from  $H_2O$ . M.p. 143°. Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Oxime* : yellow cryst. M.p. 144°.

*Dioxime* : cryst. from EtOH. M.p. 168°.

Noelting, Kadiera, *Ber.*, 1906, 39, 2059.

**2 : 4 : 6-Trihydroxybenzil.**

Colourless needles from EtOH.Aq. Darkens at 260°, m.p. 287°.  $FeCl_3 \rightarrow$  purple col. Reduces Fehling's.

*Triacetyl*: m.p. 248°.

Marsh, Stephen, *J. Chem. Soc.*, 1925, 1636.

**2 : 4 : 2'-Trihydroxybenzil.**

2'-*Me ether*:  $C_{15}H_{12}O_5$ . MW, 272. Darkens at 210°, m.p. 223°. 2 : 4-*Diacetyl*: m.p. 144°.

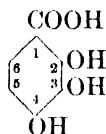
See previous reference.

**2 : 4 : 4'-Trihydroxybenzil.**

4'-*Me ether*: darkens at 225°, m.p. 234°. 2 : 4-*Diacetyl*: m.p. 178.5°.

See previous reference.

**2 : 3 : 4-Trihydroxybenzoic Acid (Pyrogallol-4-carboxylic acid)**



$C_7H_6O_5$

MW, 170

Needles +  $H_2O$  from  $H_2O$ . Decomp. at 207-8° (215-20°). Sol. EtOH. Spar. sol. Et<sub>2</sub>O. Sublimes in  $CO_2$ .  $FeCl_3 \rightarrow$  violet col. Hot  $H_2O \rightarrow$  pyrogallol.

*Me ester*:  $C_8H_8O_5$ . MW, 184. Needles +  $2\frac{1}{2}H_2O$  from  $H_2O$ . M.p. 151-2°. 4-*Me ether*:  $C_9H_{10}O_5$ . MW, 198. Needles from EtOH.Aq. M.p. 101-4°. 4-*Me ether*-2 : 3-*diacetyl*: plates from MeOH. M.p. 108°. 3 : 4-*Di-Me ether*:  $C_{10}H_{12}O_5$ . MW, 212. Needles or prisms from EtOH. M.p. 75-8°. 3 : 4-*Di-Me ether*-2-*acetyl*: cryst. from EtOH. M.p. 62-4°. 2 : 3-*Di-Me ether*-4-*benzoyl*: cryst. from EtOH.Aq. M.p. 79-80°.

*Et ester*:  $C_9H_{10}O_5$ . MW, 198. Cryst. +  $1H_2O$  from  $H_2O$ . M.p. anhyd. 102°.

*Hydrazide*: grey cryst. M.p. above 180° decomp.

4-*Me ether*:  $C_9H_8O_5$ . MW, 184. Needles from  $H_2O$ . M.p. 207-8°.  $FeCl_3 \rightarrow$  blue col.

2 : 3-*Di-Me ether*:  $C_9H_{10}O_5$ . MW, 198. Plates from  $H_2O$ . M.p. 154-5°. Insol. ligroin. Sol. MeOH, AcOEt, Me<sub>2</sub>CO.  $FeCl_3 \rightarrow$  brownish-yellow col.

3 : 4-*Di-Me ether*: needles from  $H_2O$ . M.p. 169-72°.

2 : 3 : 4-*Tri-Me ether*:  $C_{10}H_{12}O_5$ . MW, 212. Cryst. from  $H_2O$  or petrol. M.p. 99°. *Me ester*:  $C_{11}H_{14}O_5$ . MW, 226. Oil. B.p. 281°.

*Chloride*:  $C_{10}H_{11}O_4Cl$ . MW, 230.5. Cryst. from petrol. M.p. 42°. B.p. 175°/11 mm. *Amide*:  $C_{10}H_{13}O_4N$ . MW, 211. M.p. 130-1°.

2 : 3 : 4-*Tri-Et ether*:  $C_{13}H_{18}O_5$ . MW, 254. Cryst. from EtOH. M.p. 105°.

Dict. of Org. Comp.—III.

2 : 3-*Diacetyl*: needles +  $1H_2O$  from MeOH.Aq. M.p. anhyd. 157°.

2 : 3 : 4-*Triacetyl*: prisms from xylene. M.p. 164°.

4-*Benzoyl*: plates from MeOH.Aq. M.p. 210-11°.

2 : 3-*Diacetyl*-4-*benzoyl*: needles from petrol- $CHCl_3$ . M.p. 161-2°.

Hemmelmayer, *Monatsh.*, 1917, 38, 81, 88. Pacsu, *Ber.*, 1923, 56, 418.

Kostanecki, *Ber.*, 1885, 18, 3205.

**2 : 3 : 5-Trihydroxybenzoic Acid.**

2 : 3-*Di-Me ether*: cryst. M.p. 186-8°.

2 : 3 : 5-*Tri-Me ether*: plates from  $H_2O$ . M.p. 105° (141-3°).

Smith, Laforge, *J. Am. Chem. Soc.*, 1931, 53, 3074.

Faltis, Kloiber, *Monatsh.*, 1929, 53 and 54, 633.

**2 : 3 : 6-Trihydroxybenzoic Acid.**

2 : 3-*Di-Me ether*: needles from  $H_2O$ . M.p. 82°.

2 : 3 : 6-*Tri-Me ether*: cryst. from  $H_2O$ . M.p. 145-6°.

Smith, Laforge, *J. Am. Chem. Soc.*, 1931, 53, 3075.

**2 : 4 : 5-Trihydroxybenzoic Acid (Hydroxyhydroquinone-carboxylic acid).**

Needles +  $\frac{1}{2}H_2O$  from  $H_2O$ . M.p. 217-18° decomp.  $FeCl_3 \rightarrow$  blue to red col. Loses  $CO_2$  with  $H_2O$  at 100°.

4 : 5-*Di-Me ether*:  $C_9H_{10}O_5$ . MW, 198. Brown needles from  $H_2O$ . M.p. 202° decomp. *Me ester*:  $C_{10}H_{12}O_5$ . MW, 212. Needles from  $H_2O$ . M.p. 95°.

2 : 4 : 5-*Tri-Me ether*: see Asarylic Acid.

2 : 4-*Di-Me*-5-*Et ether*:  $C_{11}H_{14}O_5$ . MW, 226. Needles from  $H_2O$ , prisms from  $C_6H_6$ . M.p. 137°.

2 : 5-*Di-Me*-4-*Et ether*: prisms from  $H_2O$ . M.p. 130°.

2 : 4 : 5-*Tri-Et ether*:  $C_{13}H_{18}O_5$ . MW, 254. Needles from EtOH.Aq. M.p. 134°.

2 : 4 : 5-*Triacetyl*: plates or needles from  $C_6H_6$ . M.p. 162-3°.

Head, Robertson, *J. Chem. Soc.*, 1930, 2439.

**2 : 4 : 6-Trihydroxybenzoic Acid (Phloroglucinol-carboxylic acid).**

Cryst. +  $1H_2O$ . Decomp. on heating.  $FeCl_3 \rightarrow$  blue col. Absorbs O in alk. sol.

*Me ester*:  $C_8H_8O_5$ . MW, 184. Cryst. from EtOH.Aq. M.p. 174-6°. 4-*Me*-2-*Et ether*:

$C_{11}H_{14}O_5$ . MW, 226. Needles from EtOH. M.p. 97-9°. 2 : 4 : 6-Tri-acetyl: needles from MeOH. M.p. 77-9°.

*Et ester*:  $C_9H_{10}O_5$ . MW, 198. Prisms or needles +  $1H_2O$  from  $H_2O$ , prisms from ligroin. M.p. 129°.  $FeCl_3 \rightarrow$  violet col. 2 : 6-Di-Et ether:  $C_{13}H_{18}O_5$ . MW, 256. Prisms from MeOH. M.p. 180-1°.

4-Me ether:  $C_8H_8O_5$ . MW, 184. Grey needles from  $Et_2O-C_6H_6$ . M.p. 141° decomp. *Me ester*:  $C_9H_{10}O_5$ . MW, 198. Needles from MeOH. M.p. 114-16°.

2 : 6-Di-Me ether:  $C_9H_{10}O_5$ . MW, 198. Plates. M.p. 175° decomp. *Me ester*:  $C_{10}H_{12}O_5$ . MW, 212. Plates from MeOH.Aq. M.p. 189° decomp.

4 : 6-Di-Me ether: needles from  $Et_2O-C_6H_6$ . M.p. 152-4° decomp. *Me ester*: needles from MeOH. M.p. 107-9°.

2 : 4 : 6-Tri-Me ether:  $C_{10}H_{12}O_5$ . MW, 212. Needles from EtOH.Aq. M.p. 142-4° decomp. *Me ester*:  $C_{11}H_{14}O_5$ . MW, 226. Cryst. from MeOH. M.p. 67-70°. *Et ester*:  $C_{12}H_{16}O_5$ . MW, 240. Needles from ligroin. M.p. 77-8°.

4-Me-2 : 6-Di-Et ether:  $C_{12}H_{16}O_5$ . MW, 240. Plates from MeOH. M.p. 168°.

4-Acetyl: needles from MeOH. M.p. 177-8°.

Skraup, *Monatsh.*, 1889, 10, 724.  
Sonn, Winzer, *Ber.*, 1928, 61, 2303.

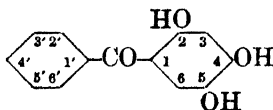
### 3 : 4 : 5-Trihydroxybenzoic Acid.

See Gallic Acid.

### 2 : 3 : 4-Trihydroxybenzophenone.

See Gallobenzophenone.

### 2 : 4 : 5-Trihydroxybenzophenone



$C_{13}H_{10}O_4$

MW, 230

5-Me ether:  $C_{14}H_{12}O_4$ . MW, 244. Yellow needles from EtOH. M.p. 183-5°.

4 : 5-Di-Me ether:  $C_{15}H_{14}O_4$ . MW, 258. Greenish-yellow cryst. from EtOH.Aq. M.p. 106-7°. 2-Acetyl: yellow needles from EtOH.Aq. M.p. 108-10°.

2 : 4 : 5-Tri-Me ether:  $C_{16}H_{16}O_4$ . MW, 272. Yellow needles from  $H_2O$ . M.p. 97°. *Phenylhydrazones*: leaflets. M.p. 178-9°.

Bargellini, Martegiani, *Atti accad. Lincei*, 1911, 20, 184.

### 2 : 4 : 6-Trihydroxybenzophenone.

See Phlorbenzophenone.

### 2 : 3 : 4'-Trihydroxybenzophenone.

Yellow needles from MeOH.Aq. M.p. 169°.  $FeCl_3 \rightarrow$  brownish-green col.

*Tri-Me ether*:  $C_{16}H_{16}O_4$ . MW, 272. Cryst. from MeOH.Aq. M.p. 86°.

Baker, Smith, *J. Chem. Soc.*, 1936, 348.

### 2 : 4 : 4'-Trihydroxybenzophenone.

Yellow needles from  $H_2O$ . M.p. 200°.  $FeCl_3 \rightarrow$  purple col.

4'-Me ether:  $C_{14}H_{12}O_4$ . MW, 244. Needles from  $H_2O$ . M.p. 165°. 2 : 4-Diacetyl: needles from EtOH.Aq. M.p. 128-30°.

*Tri-Me ether*: needles from EtOH. M.p. 73-4°.

*Triacetyl*: needles from  $H_2O$ . M.p. 96-8°.

Komarowski, Kostanecki, *Ber.*, 1894, 27, 1999.

### 2 : 6 : 2'-Trihydroxybenzophenone.

Yellow cryst. from EtOH.Aq. M.p. 133-4°. Sol. hot  $H_2O$ .  $NaOH \rightarrow$  yellow col.

Michael, *Am. Chem. J.*, 1883, 5, 89.

### 3 : 4 : 5-Trihydroxybenzophenone.

Yellow plates +  $1H_2O$ , colourless anhyd. cryst. from  $CHCl_3$ . M.p. anhyd. 177-8°. Very sol. hot  $H_2O$ . Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ . Spar. sol.  $C_6H_6$ , petrol.

Fischer, *Ber.*, 1909, 42, 1018.

### 3 : 4 : 3'-Trihydroxybenzophenone.

*Tri-Me ether*:  $C_{16}H_{16}O_4$ . MW, 272. Needles from MeOH. M.p. 83-4°. *Oxime*: prisms from EtOH. M.p. 128°.

Lea, Robinson, *J. Chem. Soc.*, 1926, 2355.

### 3 : 4 : 4'-Trihydroxybenzophenone.

*Tri-Me ether*: needles from EtOH. M.p. 98-9°.

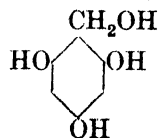
Kostanecki, Tambor, *Ber.*, 1906, 39, 4026.

### 3 : 5 : 4'-Trihydroxybenzophenone.

*Tri-Me ether*: needles from  $C_6H_6$ . M.p. 97-8°.

Mauthner, *J. prakt. Chem.*, 1913, 87, 406.

### 2 : 4 : 6-Trihydroxybenzyl Alcohol



$C_7H_8O_4$

MW, 156

2 : 4 : 6-Tri-Me ether:  $C_{10}H_{14}O_4$ . MW, 198. Cryst. from pet. ether. M.p. 63°.

Freudenberg, Harder, *Ann.*, 1927, 451, 222.

### 3 : 4 : 5-Trihydroxybenzyl Alcohol.

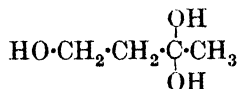
See Gallyl Alcohol.

**3 : 4 : 5-Trihydroxybenzylamine.**

See Gallylamine.

**1 : 2 : 3-Trihydroxybutane.**

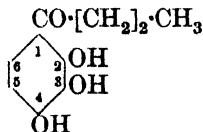
See 1-Methylglycerol.

**1 : 2 : 4-Trihydroxybutane (1 : 2 : 4-Butan-triol)** $\text{C}_4\text{H}_{10}\text{O}_3$  MW, 106Hygroscopic syrup with sweet, burning taste. B.p.  $179^\circ/13$  mm.  $D^{20}_D$  1.18.  $n^{20}_D$  1.47.4-*Me ether* :  $\text{C}_5\text{H}_{12}\text{O}_3$ . MW, 120. B.p.  $121^\circ/12$  mm.  $D^{20}_D$  1.11.  $n^{20}_D$  1.448. Di-phenylurethane : m.p.  $111-12^\circ$ .1-*Et ether* :  $\text{C}_6\text{H}_{14}\text{O}_3$ . MW, 134. Slightly yellow oil. B.p.  $210^\circ$ .4-*Et ether* : b.p.  $130^\circ/14$  mm.  $D^{20}_D$  1.08.  $n^{20}_D$  1.45. Di-phenylurethane : m.p.  $98-9^\circ$ .Tri-phenylurethane : needles from  $\text{C}_6\text{H}_6$ . M.p.  $149-52^\circ$ .1 : 4-Diacetyl : b.p.  $161-3^\circ/18$  mm.  $D^{16}_D$  1.15.  $n^{16}_D$  1.446.Triacetyl : b.p.  $150^\circ/11$  mm.  $D^{19}_D$  1.13.  $n^{19}_D$  1.436.Pariselle, *Ann. chim.*, 1911, **24**, 346.Wagner, *Ber.*, 1894, **27**, 2437.**1 : 3 : 3-Trihydroxybutane (1 : 3 : 3-Butan-triol)** $\text{C}_4\text{H}_{10}\text{O}_3$  MW, 106Tri-*Me ether* :  $\text{C}_7\text{H}_{16}\text{O}_3$ . MW, 148. B.p.  $63^\circ/20$  mm.  $D^{20}_D$  0.9398.  $n^{20}_D$  1.4112.Tri-*Et ether* :  $\text{C}_{10}\text{H}_{22}\text{O}_3$ . MW, 192. B.p.  $75^\circ/9$  mm.  $D^{20}_D$  0.8940.  $n^{20}_D$  1.4148.Tributyl ether :  $\text{C}_{16}\text{H}_{34}\text{O}_2$ . MW, 276. B.p.  $120^\circ/3$  mm.  $D^{20}_D$  0.8745.  $n^{20}_D$  1.4310.Dykstra, *J. Am. Chem. Soc.*, 1935, **57**, 2257.**1 : 2 : 3-Trihydroxybutyraldehyde.**

See Erythrose and Threose.

**1 : 2 : 3-Trihydroxybutyric Acid.**

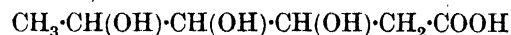
See Threonic Acid.

**2 : 3 : 4-Trihydroxybutyrophenone (4-Butyrylpyrogallol)** $\text{C}_{10}\text{H}_{12}\text{O}_4$  MW, 196Yellow needles. M.p. anhyd.  $100^\circ$ .

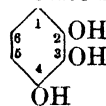
Badische, D.R.Ps., 49,149, 50,451.

**2 : 4 : 6-Trihydroxybutyrophenone.**

See Phlorbutyrophenone.

**3 : 4 : 5-Trihydroxybutyrophenone (5-Butyrylpyrogallol).**Tri-*Me ether* :  $\text{C}_{13}\text{H}_{18}\text{O}_4$ . MW, 238. Needles from MeOH. M.p.  $51-52.5^\circ$ . Sol. common org. solvents. p-Nitrophenylhydrazine : m.p.  $160^\circ$ .Bogert, Isham, *J. Am. Chem. Soc.*, 1914, **36**, 526.**2 : 3 : 4-Trihydroxycaproic Acid (Digi-toxic acid)** $\text{C}_6\text{H}_{12}\text{O}_5$  MW, 164Colourless syrup.  $\text{HNO}_3 \rightarrow$  1 : 2-dihydroxy-glutaric acid.Quinine salt : needles or columns. M.p.  $164^\circ$ . Very sol. MeOH.Aq. Spar. sol. cold  $\text{H}_2\text{O}$ .Brucine salt : columns from EtOH-Et<sub>2</sub>O. M.p.  $124^\circ$ . Very sol.  $\text{H}_2\text{O}$ . Mod. sol. EtOH.Phenylhydrazide : cryst. from EtOH. M.p.  $159^\circ$ .Zemplén, *Ber.*, 1923, **56**, 688.Kiliani, *Arch. pharm.*, 1913, **251**, 579.**2 : 3 : 4-Trihydroxycaproic Aldehyde.**

See Digitoxose.

**2 : 3 : 4-Trihydroxycinnamic Acid (Daph-netic acid)** $\text{C}_9\text{H}_8\text{O}_5$  MW, 196Tri-*Me ether* :  $\text{C}_{12}\text{H}_{14}\text{O}_5$ . MW, 238. Needles from EtOH. M.p.  $172^\circ$ .4-*Me-2 : 3-Di-Et ether* :  $\text{C}_{14}\text{H}_{18}\text{O}_5$ . MW, 266. Cryst. from pet. ether. M.p.  $157-8^\circ$ .Tri-*Et ether* :  $\text{C}_{15}\text{H}_{20}\text{O}_5$ . MW, 280. Cryst. M.p.  $193^\circ$ . Very sol. hot EtOH, Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ . Insol.  $\text{H}_2\text{O}$ ,  $\text{CS}_2$ . Ox.  $\rightarrow$  2 : 3 : 4-trimethoxybenzaldehyde and 2 : 3 : 4-trimethoxybenzoic acid.Will, Jung, *Ber.*, 1884, **17**, 1086.**2 : 4 : 5-Trihydroxycinnamic Acid (Aesc-uletic acid).**Me ester :  $\text{C}_{10}\text{H}_{10}\text{O}_5$ . MW, 210. Cryst. M.p.  $109^\circ$ .5-*Me ether* :  $\text{C}_{10}\text{H}_{10}\text{O}_5$ . MW, 210. Pale yellow needles +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $178-80^\circ$  decomp. Spar. sol. Et<sub>2</sub>O. Boiling dil. acids eliminates  $\text{CO}_2$ .Tri-*Me ether* :  $\text{C}_{13}\text{H}_{14}\text{O}_5$ . MW, 238. Needles from EtOH.Aq. M.p.  $169^\circ$ . Sol. EtOH, Et<sub>2</sub>O,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{H}_2\text{O}$ . Me ester :  $\text{C}_{13}\text{H}_{16}\text{O}_5$ .

MW, 252. Prisms from EtOH.Aq. M.p. 109°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*Tri-Et ether*: C<sub>15</sub>H<sub>20</sub>O<sub>5</sub>. MW, 280. Two forms. (i) Cryst. from EtOH. M.p. 102-3°. Conc. HCl → (ii). Ox. → triethoxybenzaldehyde and triethoxybenzoic acid. *Et ester*: C<sub>17</sub>H<sub>24</sub>O<sub>5</sub>. MW, 308. Pale yellow prisms. M.p. 51°. (ii) Cryst. from EtOH.Aq. M.p. 144°. Dist. with slight decomp. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Very spar. sol. H<sub>2</sub>O. *Et ester*: plates from EtOH. M.p. 75°.

Will, *Ber.*, 1883, 16, 2109.

Tiemann, Will, *Ber.*, 1882, 15, 2082.

### 2 : 4 : 6-Trihydroxycinnamic Acid.

*Tri-Me ether*: C<sub>12</sub>H<sub>14</sub>O<sub>5</sub>. MW, 238. Yellowish-white needles. M.p. 218° decomp. Very sol. MeOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O. *Me ester*: C<sub>13</sub>H<sub>16</sub>O<sub>5</sub>. MW, 252. Needles. M.p. 134-5°.

Herzig, Wenzel, Gehringer, *Monatsh.*, 1903, 24, 868.

### 3 : 4 : 5-Trihydroxycinnamic Acid.

Needles + 1H<sub>2</sub>O from H<sub>2</sub>O. Anhyd. at 120°. M.p. 207-8° decomp. Very sol. EtOH, Me<sub>2</sub>CO, hot H<sub>2</sub>O. Spar. sol. cold H<sub>2</sub>O, Et<sub>2</sub>O, AcOH. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, pet. ether. Sol. conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. Reduces cold NH<sub>3</sub>.AgNO<sub>3</sub>, and Fehling's on warming. KCN sol. → red → violet → yellowish-green col.

3 : 5-Di-Me ether: see Sinapic Acid.

*Tri-Me ether*: see under Sinapic Acid.

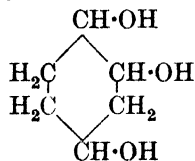
*Triacetyl*: needles from CHCl<sub>3</sub>-petrol. M.p. 168°.

Rosenmund, Boehm, *Ann.*, 1924, 437, 144.

### 1 : 2 : 3-Trihydroxycyclohexane.

See Pyrogallitol.

### 1 : 2 : 4-Trihydroxycyclohexane



C<sub>6</sub>H<sub>12</sub>O<sub>3</sub>

MW, 132

Cryst. from EtOH-Et<sub>2</sub>O. M.p. 122°.

Zelinsky, Titowa, *Ber.*, 1931, 64, 140.

### 1 : 3 : 5-Trihydroxycyclohexane.

See Phloroglucitol.

### Trihydroxydiacetylbenzene.

See Gallodiacetophenone.

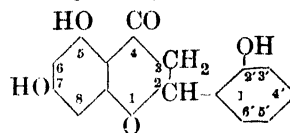
### 5 : 7 : 4'-Trihydroxy-3 : 5'-dimethoxy-flavonol.

See Syringetin.

### 1 : 1 : 1-Trihydroxyethane.

See Orthoacetic Acid.

### 5 : 7 : 2'-Trihydroxyflavanone



C<sub>15</sub>H<sub>12</sub>O<sub>5</sub>

MW, 272

Needles from AcOH.Aq. M.p. 185-7°. FeCl<sub>3</sub> → red col.

2'-Me ether: see Citronetin.

5(7)-Me ether: C<sub>16</sub>H<sub>14</sub>O<sub>5</sub>. MW, 286. Cryst. from EtOH. M.p. 192°. FeCl<sub>3</sub> → reddish-brown col.

*Tri-Me ether*: C<sub>18</sub>H<sub>18</sub>O<sub>5</sub>. MW, 314. Needles from EtOH. M.p. 124-5°. Sol. conc. H<sub>2</sub>SO<sub>4</sub> → yellowish-brown col.

Shinoda, Sato, *Chem. Zentr.*, 1931, II, 2326.

### 5 : 7 : 3'-Trihydroxyflavanone.

Plates from AcOH. M.p. 240-1°. FeCl<sub>3</sub> → violet-brown col.

3'-Me ether: C<sub>16</sub>H<sub>14</sub>O<sub>5</sub>. MW, 286. Plates. M.p. 179-80°. *Oxime*: plates from EtOH.Aq. M.p. 194-5°. 5(7)-Me ether: C<sub>17</sub>H<sub>16</sub>O<sub>5</sub>. MW, 300. Plates from EtOH. M.p. 96°. *Diacetyl*: cryst. from EtOH. M.p. 106-7°.

5(7)-Me ether: needles from EtOH. M.p. 182°.

*Acetyl*: cryst. from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 43-5°.

See previous reference.

### 5 : 7 : 4'-Trihydroxyflavanone.

See Naringenin.

### 7 : 3' : 4'-Trihydroxyflavanone.

See Butin.

### 5 : 6 : 7-Trihydroxyflavone.

See Baicalein.

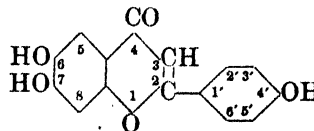
### 5 : 7 : 8-Trihydroxyflavone.

See Norwogonin.

### 5 : 7 : 4'-Trihydroxyflavone.

See Apigenin.

### 6 : 7 : 4'-Trihydroxyflavone



C<sub>15</sub>H<sub>10</sub>O<sub>5</sub>

MW, 270

Yellow needles + 1H<sub>2</sub>O from AcOH.Aq. Anhyd. at 110°. Decomp. at 300°. Sol. EtOH, NaOH.Aq. Alc. FeCl<sub>3</sub> → green col.

Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with green fluor.

*Di-Me ether*:  $\text{C}_{17}\text{H}_{14}\text{O}_5$ . MW, 298. Cryst. +  $\text{H}_2\text{O}$ . M.p. 158–62° decomp. Sol. EtOH, AcOH. Spar. sol.  $\text{C}_6\text{H}_6$ . Alc.  $\text{FeCl}_3 \rightarrow$  red col. *Acetyl*: cryst. from EtOH. M.p. 164–6°.

*Tri-Me ether*:  $\text{C}_{18}\text{H}_{16}\text{O}_5$ . MW, 312. Needles from EtOH. M.p. 184–6°. Sol. AcOH,  $\text{C}_6\text{H}_6$ . Fluor. sol. in EtOH. Sol. conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with green fluor.

*Triacetyl*: needles from AcOH. M.p. 234–6°.

Bargellini, Grippa, Gazz. chim. ital., 1927, 57, 605.

### 7 : 8 : 4'-Trihydroxyflavone.

Yellow needles from EtOH.Aq. Sinters at 279°, m.p. 299–300° decomp. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. Alc.  $\text{FeCl}_3 \rightarrow$  green col. NaOH.Aq.  $\rightarrow$  orange sol.

*Tri-Me ether*: yellow needles from EtOH. M.p. 189–90°.

*Triacetyl*: white needles. M.p. 183°.

Badhwar, Kang, Venkataraman, J. Chem. Soc., 1932, 1109.

### 7 : 3' : 4'-Trihydroxyflavone.

*3' : 4'-Di-Me ether*:  $\text{C}_{17}\text{H}_{14}\text{O}_5$ . MW, 298. Plates from AcOH.Aq. M.p. 255°. Sol. EtOH with violet fluor. No col. with  $\text{FeCl}_3$ .

Baker, J. Chem. Soc., 1933, 1387.

### 3' : 4' : 5'-Trihydroxyflavone.

Pale yellow needles from EtOH.Aq. Does not melt below 280°. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. NaOH  $\rightarrow$  red sol.  $\text{FeCl}_3 \rightarrow$  green ppt.

*Tri-Me ether*:  $\text{C}_{18}\text{H}_{16}\text{O}_5$ . MW, 312. Needles from EtOH.Aq. M.p. 174–5°.

*Triacetyl*: prisms from EtOH. M.p. 195–6°.

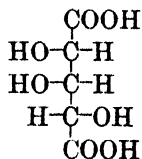
Hattori, Chem. Zentr., 1932, II, 710.

### Trihydroxyflavonol.

See Tetrahydroxyflavone.

### Trihydroxyglutaric Acid

#### I. Arabo-trihydroxyglutaric Acid.



$\text{C}_5\text{H}_8\text{O}_7$

MW, 180

*d.*

Plates from  $\text{H}_2\text{O}$ , cryst. from  $\text{Me}_2\text{CO}$ . M.p. 128°. Very sol.  $\text{H}_2\text{O}$ . Sol. EtOH, hot  $\text{Me}_2\text{CO}$ .  $[\alpha]_D^{20} + 22.2^\circ$  in  $\text{H}_2\text{O}$ .

*Di-Me ester*:  $\text{C}_7\text{H}_{12}\text{O}_7$ . MW, 208. *Tri-Me*

*ether*:  $\text{C}_{10}\text{H}_{18}\text{O}_7$ . MW, 250. B.p. about 143°/15 mm., 100°/0.1 mm.  $n_D^{21} 1.4353$ .  $[\alpha]_D^{20} - 47.5^\circ$  in MeOH,  $[\alpha]_D^{20} - 42.5^\circ$  in  $\text{H}_2\text{O}$ .

*Diamide*:  $\text{C}_5\text{H}_{10}\text{O}_5\text{N}_2$ . MW, 178. *Tri-Me ether*:  $\text{C}_8\text{H}_{16}\text{O}_5\text{N}_2$ . MW, 220. M.p. 232–3° decomp.  $[\alpha]_D^{20} - 49^\circ$  in  $\text{H}_2\text{O}$ .

*l.*

Plates from EtOH. M.p. 127°.  $[\alpha]_D^{18} - 23.3^\circ$  in  $\text{H}_2\text{O}$ .  $k = 1.32 \times 10^{-3}$  at 25°. *Ag salt*: m.p. 173°. *Quinine salt*: needles + 5 $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. 180°.  $[\alpha]_D - 112.5^\circ$ . *Brucine salt*: needles from EtOH.Aq. M.p. 175–6°.  $[\alpha]_D - 41.67^\circ$ .

*Di-Me ester*: *tri-Me ether*, syrup. B.p. 143°/18 mm., 74–6°/0.005 mm. Sol.  $\text{H}_2\text{O}$  and most org. solvents.  $n_D^{20} 1.4350$ .  $[\alpha]_D + 47.3^\circ$  in MeOH,  $[\alpha]_D + 45^\circ$  in  $\text{H}_2\text{O}$ .

*Diamide*: *tri-Me ether*, prisms from MeOH. M.p. 232–3°.  $[\alpha]_D + 50.0^\circ$  in  $\text{H}_2\text{O}$ .

*dl.*

Cryst. from  $\text{Me}_2\text{CO}$ . M.p. 154.5° decomp. Very sol.  $\text{H}_2\text{O}$ , EtOH. Sol.  $\text{Me}_2\text{CO}$ .  $k = 6.9 \times 10^{-4}$  at 25°.

Hirst, Smith, J. Chem. Soc., 1928, 3153.

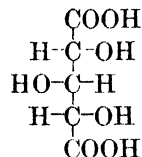
Votocěk, Ber., 1910, 43, 472.

Nef, Ann., 1914, 403, 252.

Ruff, Ber., 1899, 32, 558.

Haworth, Hirst, Jones, J. Chem. Soc., 1927, 2428.

#### II. Xylo-trihydroxyglutaric Acid.



Cryst. from AcOEt. M.p. 152° decomp. Very sol.  $\text{H}_2\text{O}$ , hot EtOH. Spar. sol. hot  $\text{Me}_2\text{CO}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Optically inactive. Heat of comb.  $\text{C}_7$  389.5 Cal.  $k = 6.6 \times 10^{-4}$  at 25°. Evap. aq. sol.  $\rightarrow$  lactone. HI(+P)  $\rightarrow$  glutaric acid. Reduces  $\text{NH}_3\text{AgNO}_3$  but not Fehling's.

*Di-phenylhydrazide*: plates. Sinters at 175°, decomp. at 210°.

*Di-Me ester*: 1 : 2-*Di-Me ether*.  $\text{C}_9\text{H}_{16}\text{O}_7$ . MW, 236. Oil. B.p. 132°/12 mm. 97–9°/0.003 mm. Hyd.  $\rightarrow$  lactone of 1 : 2-dimethoxyglutaric acid.

*Diamide*: *tri-Me ether*, m.p. 195–8° (194–5°).

Schmidt, Zeiser, Ber., 1934, 67, 2124.

Fischer, Herborn, Ber., 1896, 29, 1965.

Haworth, Jones, J. Chem. Soc., 1927, 2349.

Hirst, J. Chem. Soc., 1926, 350.

**Trihydroxyheptane.**

See Heptantriol.

**Trihydroxyhexane.**

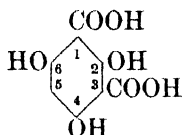
See Hexantriol.

**2 : 4 : 6-Trihydroxyisobutyrophenone.**

See Phlorisobutyrophenone.

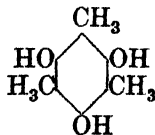
**5 : 7 : 4'-Trihydroxyisoflavone.**

See Genistein.

**2 : 4 : 6-Trihydroxyisophthalic Acid**  
(Phloroglucinol-dicarboxylic acid) $C_6H_6O_7$ 

MW, 214

Free acid unknown.

*Di-Me ester*:  $C_{10}H_{10}O_7$ . MW, 242. Needles or prisms from MeOH. M.p. 145–6°.*Di-Et ester*:  $C_{12}H_{14}O_7$ . MW, 270. Needles from EtOH. M.p. 104° (107–8°). *Triacetyl*: prisms from EtOH. M.p. 96–8°.*Tri-Me ether*:  $C_{11}H_{12}O_7$ . MW, 256. Prisms from  $H_2O$ . Sinters at 260°. *Di-Me ester*:  $C_{13}H_{16}O_7$ . MW, 284. Plates from  $Et_2O$ . M.p. 120–1°. Sol.  $CHCl_3$ ,  $Me_2CO$ ,  $AcOEt$ ,  $AcOH$ ,  $C_6H_6$ . Mod. sol. MeOH,  $Et_2O$ . Spar. sol. ligroin. *Di-Et ester*:  $C_{15}H_{20}O_7$ . MW, 312. Plates from EtOH. M.p. 90–1°. Sol. EtOH,  $Et_2O$ . Spar. sol. pet. ether.Leuchs, *Ann.*, 1928, 460, 1.Leuchs, Dzieng, *Ann.*, 1924, 440, 151.Leuchs, Simion, *Ber.*, 1911, 44, 1878.**4 : 5 : 6-Trihydroxyisophthalic Acid**  
(Gallocarboxylic acid, pyrogallol-4 : 6-dicarboxylic acid).Needles +  $3H_2O$  from  $H_2O$ . M.p. 283° decomp. Hot  $Ac_2O \rightarrow$  pyrogallol triacetate.*Tri-Me ether*: prisms from MeOH. M.p. 191°. *Di-Me ester*: plates from MeOH. M.p. 35–6°.Feist, Awe, *Ber.*, 1926, 59, 175.**2 : 4 : 6-Trihydroxymesitylene** (2 : 4 : 6-Trimethylphloroglucinol) $C_9H_{12}O_3$ 

MW, 168

Needles from AcOH. M.p. 184°. Sol. MeOH, EtOH,  $AcOEt$ , hot  $H_2O$ . Spar. sol. $C_6H_6$ , hot pet. ether. Reduces  $NH_3 \cdot AgNO_3$ .  $FeCl_3 \rightarrow$  reddish-violet col.*Mono-Me ether*:  $C_{10}H_{14}O_3$ . MW, 182. Needles from  $H_2O$  or  $C_6H_6$ . M.p. 121°. B.p. 196–8°/20 mm. *Diacetyl*: cryst. from MeOH. Aq. M.p. 66–8°.*Mono-Et ether*:  $C_{11}H_{16}O_3$ . MW, 196. Cryst. from  $CHCl_3$ . M.p. 130°.*Triacetyl*: prisms from  $C_6H_6$ . M.p. 165–7°.Weidel, Wenzel, *Monatsh.*, 1898, 19, 257.**Trihydroxymethane.**

See Orthoformic Acid.

**5 : 7 : 3'-Trihydroxy-4'-methoxyflavone.**

See Hesperetin.

**5 : 7 : 4'-Trihydroxy-3'-methoxyflavone.**

See Homoeriodictyol.

**3 : 5 : 7-Trihydroxy-4'-methoxyflavone.**

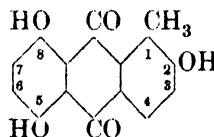
See Kaempferide.

**5 : 7 : 3'-Trihydroxy-4'-methoxyflavone.**

See Diosmetin.

**5 : 7 : 4'-Trihydroxy-3'-methoxyflavone.**

See Chrysoeriol.

**2 : 5 : 8-Trihydroxy-1-methylantranthraquinone** $C_{15}H_{10}O_5$ 

MW, 270

Dark red needles from  $CHCl_3$ . M.p. 270°. Sol. alkalis and conc.  $H_2SO_4$  with bluish-red col.*2-Me ether*:  $C_{16}H_{12}O_5$ . MW, 284. Dark red needles from AcOH. M.p. 249–249.5°. Sol. conc.  $H_2SO_4$  with bluish-violet col., in alkalis with bluish-red col.Gardner, Adams, *J. Am. Chem. Soc.*, 1923, 45, 2455.Graves, Adams, *ibid.*, 2439.**3 : 5 : 8-Trihydroxy-1-methylantranthraquinone.**

Red needles from EtOH. M.p. 260°.

*Triacetyl*: yellow needles from  $C_6H_6$ . M.p. 179°.Dimroth, Fick, *Ann.*, 1916, 411, 330.**3 : 7 : 8-Trihydroxy-1-methylantranthraquinone.**

See 8-Methylflavopurpurin.

**4 : 5 : 8-Trihydroxy-1-methylantranthraquinone.**

Red needles from AcOH. M.p. 276–8°. Mod. sol. cold EtOH. Sublimes. Sol. alkalis

**4 : 7 : 8-Trihydroxy-1-methylanthra-quinone**

with violet-red col., in conc.  $\text{H}_2\text{SO}_4$  with blue to bluish-violet col. Alc.  $\text{FeCl}_3 \rightarrow$  greenish-brown col.

**5 : 8-Di-Me ether** :  $\text{C}_{17}\text{H}_{14}\text{O}_5$ . MW, 298. Red needles from  $\text{Me}_2\text{CO}$ . M.p.  $224^\circ$ . Sol. alkalis with red col., in conc.  $\text{H}_2\text{SO}_4$  with blue to bluish-red col.

**Tri-Me ether** :  $\text{C}_{18}\text{H}_{16}\text{O}_5$ . MW, 312. Yellow needles from EtOH. M.p.  $249-250.5^\circ$ .

**Triacetyl** : m.p.  $197^\circ$ .

Keimatsu, Hirano, Yoshimi, *Chem. Zentr.*, 1930, II, 2384.

Gardner, Adams, *J. Am. Chem. Soc.*, 1923, **45**, 2455.

Graves, Adams, *ibid.*, 2439.

**4 : 7 : 8 - Trihydroxy - 1 - methylanthra - quinone.**

Red needles from AcOH. M.p.  $301^\circ$  (decomp. at  $290^\circ$ ).

**7 : 8-Di-Me ether** : yellow needles from AcOEt. M.p.  $168-9^\circ$ . **Acetyl** : yellow needles from EtOH. M.p.  $173-4^\circ$ .

**Triacetyl** : yellow needles from EtOH. M.p.  $204-5^\circ$ .

Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 2011.

**5 : 6 : 7 - Trihydroxy - 1 - methylanthra - quinone.**

See 5-Methylanthragallol.

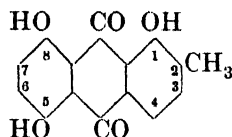
**6 : 7 : 8 - Trihydroxy - 1 - methylanthra - quinone.**

See 8-Methylanthragallol.

**1 : 5 : 6 - Trihydroxy - 2 - methylanthra - quinone.**

See Morindone.

**1 : 5 : 8 - Trihydroxy - 2 - methylanthra - quinone**



$\text{C}_{15}\text{H}_{10}\text{O}_5$

MW, 270

Red needles from AcOH. M.p.  $253-4^\circ$ . Very sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , toluene. Mod. sol. EtOH. Sublimes at  $250-60^\circ$ . Sol. alkalis with red col., in conc.  $\text{H}_2\text{SO}_4$  with bluish-red col. Alc.  $\text{FeCl}_3 \rightarrow$  red col.

**5 : 8-Di-Me ether** :  $\text{C}_{17}\text{H}_{14}\text{O}_5$ . MW, 298. Red needles from AcOH. M.p.  $165^\circ$ . Sol. alkalis with red col., in conc.  $\text{H}_2\text{SO}_4$  with blue to bluish-red col.

**Tri-Me ether** :  $\text{C}_{18}\text{H}_{16}\text{O}_5$ . MW, 312. Red needles from EtOH. M.p.  $206.5-207^\circ$ .

**839 5 : 7 : 4'-Trihydroxy-2-methylisoflavone**

**Triacetyl** : yellow needles. M.p.  $215^\circ$ .

Keimatsu, Hirano, *Chem. Zentr.*, 1930, II, 1551.

Graves, Adams, *J. Am. Chem. Soc.*, 1923, **45**, 2439.

**1 : 7 : 8 - Trihydroxy - 2 - methylanthra - quinone.**

Brownish-yellow needles from AcOH.Aq. M.p.  $287-8^\circ$ . Sol. NaOH with red col., in conc.  $\text{H}_2\text{SO}_4$  with bluish-red col.

**Tri-Me ether** : yellow needles from AcOEt. M.p.  $209-10^\circ$ . Sol. conc.  $\text{H}_2\text{SO}_4$  with reddish-purple col.

Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 288.

Simonsen, *J. Chem. Soc.*, 1924, 125, 726.

**3 : 5 : 6 - Trihydroxy - 2 - methylanthra - quinone.**

Orange-red needles from  $\text{PhNO}_2$ . Does not melt below  $330^\circ$ .

**5 : 6-Di-Me ether** :  $\text{C}_{17}\text{H}_{14}\text{O}_5$ . MW, 298. Yellow needles from AcOH. M.p. about  $310^\circ$ .

**Triacetyl** : yellow needles from EtOH. M.p.  $232-3^\circ$ .

Jacobson, Adams, *J. Am. Chem. Soc.*, 1925, **47**, 2011.

**3 : 5 : 8 - Trihydroxy - 2 - methylanthra - quinone.**

See Rhababerone.

**3 : 7 : 8 - Trihydroxy - 2 - methylanthra - quinone.**

See 7-Methylflavopurpurin.

**4 : 5 : 6 - Trihydroxy - 2 - methylanthra - quinone.**

**Tri-Me ether** : needles from AcOH.Aq. M.p.  $164-5^\circ$ . Sol. conc.  $\text{H}_2\text{SO}_4$  with reddish-purple col.

Simonsen, *J. Chem. Soc.*, 1924, 125, 724.

**4 : 5 : 7 - Trihydroxy - 2 - methylanthra - quinone.**

See Frangula-emodin.

**4 : 5 : 8 - Trihydroxy - 2 - methylanthra - quinone.**

See Helminthosporin.

**5 : 6 : 7 - Trihydroxy - 2 - methylanthra - quinone.**

See 6-Methylanthragallol.

**6 : 7 : 8 - Trihydroxy - 2 - methylanthra - quinone.**

See 7-Methylanthragallol.

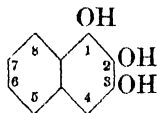
**5 : 7 : 4'-Trihydroxy-2-methylisoflavone.**

See 2-Methylgenistein.



**1 : 4 : 5-Trihydroxy-2-methylxanthone.**

See Ravenelin.

**1 : 2 : 3-Trihydroxynaphthalene** (*Naphthopyrogallol*) $C_{10}H_8O_3$ 

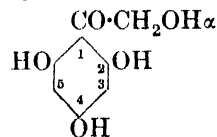
MW, 176

Prisms. Decomp. above  $250^\circ$ . Sol.  $H_2O$ , EtOH,  $Me_2CO$ .*Triacetyl*: prisms from AcOH. M.p.  $250-5^\circ$ .Zincke, Noack, *Ann.*, 1897, **295**, 17.**1 : 2 : 4-Trihydroxynaphthalene.**Needles from  $C_6H_6$ . M.p.  $154^\circ$ . Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol.  $C_6H_6$ .*Triacetyl*: plates from EtOH.Aq. M.p.  $134-5^\circ$ .Bayer, D.R.P., 101,607, (*Chem. Zentr.*, 1899, I, 1094).Thiele, Winter, *Ann.*, 1911, **311**, 345.**1 : 3 : 6-Trihydroxynaphthalene.**Cryst. from  $H_2O$ . M.p.  $95^\circ$ . Sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $Me_2CO$ . Spar. sol.  $CHCl_3$ ,  $C_6H_6$ , pet. ether.  $FeCl_3 \rightarrow$  yellowish-brown col.*Triacetyl*: needles from EtOH.Aq. M.p.  $112-13^\circ$ .Meyer, Hartmann, *Ber.*, 1905, **38**, 3950.**1 : 4 : 5-Trihydroxynaphthalene.**See  $\alpha$ -Hydrojuglone.**1 : 4 : 6-Trihydroxynaphthalene.**Needles from  $Et_2O$ -pet. ether. M.p.  $138-40^\circ$ . Very unstable in moist air.*Triacetyl*: needles from EtOH. M.p.  $94-5^\circ$ .Fischer, Bauer, *J. prakt. Chem.*, 1916, **94**, 8.**1 : 6 : 7-Trihydroxynaphthalene.**Yellow needles from xylene. M.p.  $177^\circ$ . Sol. EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ .  $FeCl_3 \rightarrow$  blue col.*Tri-Me ether*:  $C_{13}H_{14}O_3$ . MW, 218. Prisms from ligroin. M.p.  $127-8^\circ$ .*Triacetyl*: greenish cryst. from EtOH.Aq. M.p.  $143-4^\circ$ .Friedländer, Silberstern, *Monatsh.*, 1902, **23**, 530.**5 : 6 : 8-Trihydroxy-1 : 4-naphthoquinone.**

See Naphthopurpurin.

**Trihydroxypentane.**

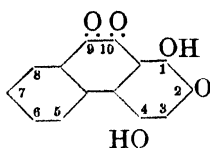
See Pentantriol.

**2 : 4 : 6 - Trihydroxyphenacyl Alcohol**(2 : 4 : 6 :  $\alpha$ -Tetrahydroxyacetophenone) $C_8H_8O_5$ 

MW, 184

 $\alpha$ -Me ether:  $C_9H_{10}O_5$ . MW, 198. Needles from hot  $H_2O$ . M.p.  $192^\circ$ .  $FeCl_3 \rightarrow$  violet col.4 : 6 :  $\alpha$ -Tri-Me ether:  $C_{11}H_{14}O_5$ . MW, 226. Needles from EtOH. M.p.  $102-4^\circ$ . Oxime: needles from MeOH.Aq. M.p.  $147-9^\circ$ .*Tetra-Me ether*:  $C_{12}H_{16}O_5$ . MW, 240. Cryst. from MeOH. M.p.  $50^\circ$ . No col. with  $FeCl_3$ .4 : 6 :  $\alpha$ -Tri-Et ether:  $C_{14}H_{20}O_5$ . MW, 268. Needles from EtOH. M.p.  $96-7^\circ$ .Herzig, Hoffmann, *Ber.*, 1909, **42**, 156.Perkin, *J. Chem. Soc.*, 1911, **99**, 1724.Slater, Stephen, *J. Chem. Soc.*, 1920, **117**, 316.**3 : 4 : 5 - Trihydroxyphenacyl Alcohol**(3 : 4 : 5 :  $\alpha$ -Tetrahydroxyacetophenone). $\alpha$ -Me ether: *triacetyl*, cryst. from EtOH. M.p.  $132-3^\circ$ .3 : 5-Di-Me ether:  $C_{10}H_{12}O_5$ . MW, 212. Needles +  $H_2O$  from boiling  $H_2O$ . M.p.  $93-5^\circ$ , anhyd.  $132^\circ$ . Reduces cold Fehling's. Alc.  $FeCl_3 \rightarrow$  weak olive-green col. *Diacetyl*: prismatic needles from MeOH. M.p.  $123^\circ$ . 4-Benzoyl: prismatic needles from 50% EtOH. M.p.  $173-5^\circ$ . *Dibenzoyl*: micro-needles from  $C_6H_6$ -pet. ether. M.p.  $128^\circ$ .3 : 4 : 5-Tri-Me ether:  $\alpha$ -acetyl, m.p.  $88^\circ$ .*Tetra-Me ether*: needles from  $C_6H_6$ -pet. ether. M.p.  $54^\circ$ . B.p.  $212^\circ/15$  mm. *Semicarbazone*: needles from EtOH.Aq. M.p.  $158^\circ$ .3 : 4 : 5-Triacetyl: needles from  $C_6H_6$ . M.p.  $87-8^\circ$ . Sol.  $Et_2O$ ,  $Me_2CO$ , AcOEt,  $C_6H_6$ , hot EtOH,  $C_6H_6$ . Spar. sol.  $H_2O$ . Insol. pet. ether. No col. with  $FeCl_3$ . Reduces cold Fehling's.*Tetra-acetyl*: cryst. from EtOH. M.p.  $124-5^\circ$ .3 : 4 : 5-Tribenzoyl:  $\alpha$ -formyl, prismatic needles from formic acid. M.p.  $116-17^\circ$ .  $\alpha$ -Acetyl: pale yellow plates from EtOH. M.p.  $138-40^\circ$ .  $\alpha$ -p-Toluenesulphonyl: needles. M.p.  $148^\circ$ .Pratt, Robinson, *J. Chem. Soc.*, 1925, 173.Levy, Posternack, Robinson, *J. Chem. Soc.*, 1931, 2705.Reynolds, Robinson, *J. Chem. Soc.*, 1934, 1040.

## 1 : 2 : 4-Trihydroxyphenanthraquinone

 $C_{14}H_8O_5$ 

MW, 256

Very readily oxidised in air. Very sol. EtOH. Spar. sol. most other org. solvents. Does not crystallise, and decomposes on heating. Sol. alkalis with green col.  $\rightarrow$  pale red in air. Sol. conc.  $H_2SO_4$  with green col., in Py with cornflower-blue col.

*Triacetyl*: orange plates from toluene. M.p. 227–8° decomp.

Fieser, *J. Am. Chem. Soc.*, 1929, 51, 1939.

## 1 : 3 : 4-Trihydroxyphenanthraquinone.

Dark red cryst. from EtOH. Very unstable to air. Spar. sol.  $H_2O$ . Sol. alkalis with green col.  $\rightarrow$  pink in air. Sol. conc.  $H_2SO_4$  with red col., in Py with red col.  $\rightarrow$  green on dilution with  $H_2O$ .

*Triacetyl*: yellow needles from AcOH. Decomp. at 240°.

Fieser, *J. Am. Chem. Soc.*, 1929, 51, 1940.

## 2 : 3 : 4-Trihydroxyphenanthraquinone.

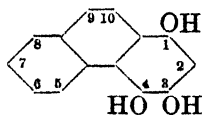
Brownish-red powder. M.p. 185° decomp.

*Monosemicarbazone*: brownish-red powder from EtOH. Decomp. at 270°.

*Phenazine*:  $C_{20}H_{12}O_3N_2$ . Brown microcryst. from EtOH. M.p. 255° decomp.

Schmidt, Schairer, *Ber.*, 1923, 56, 1337.

## 1 : 3 : 4-Trihydroxyphenanthrene

 $C_{14}H_{10}O_3$ 

MW, 226

*Triacetyl*: needles from  $C_6H_6$ -pet. ether. M.p. 138°.

Fieser, *J. Am. Chem. Soc.*, 1929, 51, 1940.

## 1 : 5 : 6-Trihydroxyphenanthrene.

1 : 5-Di-*Me ether*:  $C_{16}H_{14}O_3$ . MW, 254. Reddish-brown plates from EtOH.Aq. M.p. 164–5°. *Acetyl*: prisms from AcOH. M.p. 96–7°.

5 : 6-Di-*Me ether*: prisms from EtOH. M.p. 182–3°. 1-*Et ether*:  $C_{18}H_{16}O_3$ . MW, 282. Plates from MeOH. M.p. 100°.

*Tri-Me ether*:  $C_{17}H_{16}O_3$ . MW, 268. Plates from MeOH. M.p. 138°.

Pschorr, *Ber.*, 1912, 45, 2220; 1900, 33, 181.

## 3 : 4 : 5-Trihydroxyphenanthrene.

Plates from  $H_2O$ . M.p. 148°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

*Tri-Me ether*: cryst. from MeOH. M.p. 90°. *Picrate*: red plates from MeOH. M.p. 166°.

Pschorr, *Ann.*, 1912, 391, 53.

## 3 : 4 : 6-Trihydroxyphenanthrene.

3 : 6-Di-*Me ether*: see Thebaol.

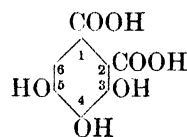
*Tri-Me ether*: oil. *Picrate*: red needles from EtOH. M.p. 109–10° (110–12°).

Pschorr, Seydel, Stöhrer, *Ber.*, 1902, 35, 4406.

Vongerichten, *Ber.*, 1902, 35, 4411.

## Trihydroxyphenyl hydroxystyryl Ketone.

See Tetrahydroxychalkone.

3 : 4 : 5-Trihydroxyphthalic Acid (*Pyrogallol-4 : 5-dicarboxylic acid*) $C_8H_6O_7$ 

MW, 214

3 : 5-Di-*Me ether*:  $C_{10}H_{10}O_7$ . MW, 242. Cryst. from  $H_2O$ . M.p. 225–7°. *Anhydride*:  $C_{10}H_8O_6$ . MW, 224. Needles from toluene. M.p. 177–9°.

*Tri-Me ether*:  $C_{11}H_{12}O_7$ . MW, 256. Plates from  $H_2O$ . M.p. 176–7° decomp.  $FeCl_3 \rightarrow$  yellow col. 1-*Me ester*:  $C_{12}H_{14}O_7$ . MW, 270. Needles from MeOH. M.p. 138–41°. *Di-Me ester*:  $C_{13}H_{16}O_7$ . MW, 284. Cryst. M.p. 64–5°. *Anhydride*:  $C_{11}H_{10}O_6$ . MW, 238. Needles from  $C_6H_6$ . M.p. 147°.

Alimchandani, Meldrum, *J. Chem. Soc.*, 1920, 117, 964.

Feist, Dschu, *Chem. Zentr.*, 1927, II, 58.

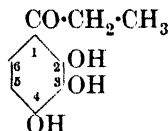
## 3 : 4 : 6-Trihydroxyphthalic Acid.

*Tri-Me ether*: prisms +  $1H_2O$  from  $H_2O$ . M.p. 216–17° (185°).

Faltis, Kloiber, Gutlohn, Attia, *Monatsh.*, 1929, 53 and 54, 632.

## Trihydroxypropane.

See Glycerol and Orthopropionic Acid.

**2 : 3 : 4-Trihydroxypropiophenone** (4-*Propionylpyrogallol*) $C_9H_{10}O_4$ 

MW, 182

Yellow needles. M.p. 127°.

Badische, D.R.P., 42,149.

**2 : 4 : 5-Trihydroxypropiophenone.**

4 : 5-*Di-Me ether* :  $C_{11}H_{14}O_4$ . MW, 210. Needles from EtOH.Aq. M.p. 124-6°.  $FeCl_3$  → green col. *Acetyl* : needles from EtOH.Aq. M.p. 117-18°. *Benzoyl* : needles. M.p. 110-11°.

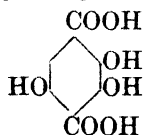
*Tri-Me ether* :  $C_{13}H_{16}O_4$ . MW, 224. Needles from  $H_2O$ . M.p. 106-8°. *Oxime* : plates from EtOH.Aq. M.p. 106-8°. *Semicarbazone* : (i) plates from EtOH.Aq. M.p. 182. (ii) Prisms from  $H_2O$ . M.p. 166-7°.

Bargellini, Martegiani, *Gazz. chim. ital.*, 1911, 41, 449.**2 : 4 : 6-Trihydroxypropiophenone.**

See Phloropropiophenone.

**3 : 4 : 5-Trihydroxypropiophenone.**

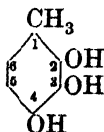
*Tri-Me ether* : needles from ligroin. M.p. 51-2°. Sol. EtOH, AcOH, warm ligroin. *p-Nitrophenylhydrazone* : red needles from EtOH. M.p. 182-3°.

Mauthner, *J. prakt. Chem.*, 1926, 112, 269.**2 : 3 : 5-Trihydroxyterephthalic Acid** $C_8H_6O_7$ 

MW, 214

Orange-yellow prisms from AcOH. M.p. 247° decomp. Sol. common org. solvents.

*Di-Et ester* :  $C_{12}H_{14}O_7$ . MW, 270. Yellow needles from  $Me_2CO$ . M.p. 116°. *Triacetyl* : cryst. from  $Me_2CO$ .Aq. M.p. 100°.

Liebermann, Lisser, *Ann.*, 1934, 513, 184.**2 : 3 : 4-Trihydroxytoluene** (4-*Methylpyrogallol*) $C_7H_8O_3$ 

MW, 140

Needles. M.p. 140-1°.

Majima, Okazaki, *Ber.*, 1916, 49, 1492.**2 : 3 : 5-Trihydroxytoluene.**

3-*Me ether* :  $C_8H_{10}O_3$ . MW, 154. Needles. M.p. 128-9°. Sol. EtOH,  $Et_2O$ , AcOH. Spar. sol. ligroin. Reduces Fehling's.

2 : 3-*Di-Me ether* :  $C_9H_{12}O_3$ . MW, 168. Plates from  $C_6H_6$ . M.p. 140-1°.

Henrich, Nachtigall, *Ber.*, 1903, 36, 894.**2 : 3 : 6-Trihydroxytoluene.**

2-*Me ether* : needles from ligroin. M.p. 117-18°.

Majima, Okazaki, *Ber.*, 1916, 49, 1490.**2 : 4 : 5-Trihydroxytoluene.**

Prisms from  $C_6H_6$ . M.p. 131-2°. Sol.  $H_2O$ . Aq. sol. oxidises readily.

4-*Me ether* :  $C_8H_{10}O_3$ . MW, 154. Needles from  $H_2O$ . M.p. 124°. Sol. common org. solvents, hot  $H_2O$ .

4-*Et ether* :  $C_9H_{12}O_3$ . MW, 168. Tablets from  $C_6H_6$ . M.p. 131°. Sublimes.

*Tri-Me ether* :  $C_{10}H_{16}O_3$ . MW, 182. Plates from MeOH.Aq. M.p. 55°. Sol.  $Et_2O$ , EtOH, pet. ether.

*Triacetyl* : cryst. from EtOH. M.p. 114-15°.

Thiele, Winter, *Ann.*, 1900, 311, 349.Luff, Perkin, Robinson, *J. Chem. Soc.*, 1910, 97, 1137.Bayer, D.R.P., 101,607, (*Chem. Zentr.*, 1899, I, 1094).**2 : 4 : 6-Trihydroxytoluene** (2-*Methylphloroglucinol*).

Needles from AcOEt. M.p. 214-16°. Sol.  $H_2O$ , EtOH,  $Et_2O$ , AcOEt, hot AcOH. Insol.  $C_6H_6$ , pet. ether. Sublimes.

2-*Me ether* :  $C_8H_{10}O_3$ . MW, 154. Prisms +  $1H_2O$  from  $H_2O$ . M.p. 91°, anhyd. 117-19°. Sol. EtOH,  $Et_2O$ , hot  $H_2O$ , hot  $C_6H_6$ .

4-*Me ether* : needles from xylene. M.p. 124°. B.p. 195-8°/20 mm. Sol. EtOH, AcOEt. Spar. sol.  $H_2O$ , ligroin.

2 : 4-*Di-Me ether* :  $C_9H_{12}O_3$ . MW, 168. Needles from xylene-ligroin. M.p. 60-1°. B.p. 178-80°/20 mm. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ , ligroin. 6-*Et ether* :  $C_{11}H_{16}O_3$ . MW, 196. Cryst. from 75% EtOH. M.p. 38°. B.p. 149-51°/16 mm.

2 : 4 : 6-*Tri-Me ether* :  $C_{10}H_{14}O_3$ . MW, 182. Cryst. M.p. 10-13°. B.p. 140-2°/18 mm.

4-*Et ether* :  $C_9H_{12}O_3$ . MW, 168. Needles from  $C_6H_6$ . M.p. 136-7°. B.p. 195-200°/13 mm. Sol. EtOH,  $Et_2O$ . *Diacetyl* : plates from EtOH. M.p. 91°.

*Triacetyl*: needles from ligroin. M.p. 76° (52°).

Herzig, Wenzel, *Monatsh.*, 1916, **37**, 573.

Boehm, *Ann.*, 1901, **318**, 286.

Herzig, Theuer, *Monatsh.*, 1900, **21**, 855.

**3 : 4 : 5-Trihydroxytoluene** (5-*Methylpyrogallol*).

Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 120°. Sublimes in needles.

3 : 5-*Di-Me ether*: cryst. from EtOH. M.p. 36°. B.p. 265°, 145-6°/12 mm. Sol. H<sub>2</sub>O, Et<sub>2</sub>O.

3 : 4-*Di-Me ether*: see Iridol.

*Tri-Me ether*: see under Iridol.

*Triacetyl*: cryst. M.p. 99°.

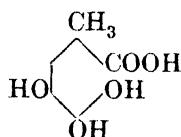
Rosauer, *Monatsh.*, 1898, **19**, 565.

Hofmann, *Ber.*, 1879, **12**, 1376.

ω-**Trihydroxytoluene**.

See Orthobenzoic Acid.

**3 : 4 : 5-Trihydroxy-o-toluic Acid**



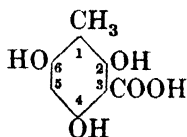
C<sub>8</sub>H<sub>8</sub>O<sub>5</sub>

MW, 184

*Me ester*: C<sub>9</sub>H<sub>10</sub>O<sub>5</sub>. MW, 198. Cryst. M.p. 155°.

Koller, Hamburg, *Monatsh.*, 1935, **65**, 373.

**2 : 4 : 6-Trihydroxy-m-toluic Acid** (4-*Methylphloroglucinol-2-carboxylic acid*)



C<sub>8</sub>H<sub>8</sub>O<sub>5</sub>

MW, 184

M.p. 177°.

4-*Me ether*: C<sub>9</sub>H<sub>10</sub>O<sub>5</sub>. MW, 198. Cryst. from Et<sub>2</sub>O-C<sub>6</sub>H<sub>6</sub>. M.p. 147° decomp. Sol. EtOH. Insol. H<sub>2</sub>O, ligroin. *Me ester*: C<sub>10</sub>H<sub>12</sub>O<sub>5</sub>. MW, 212. Cryst. from EtOH. M.p. 132-3°. Spar. sol. EtOH. *Diacetyl*: cryst. from EtOH. M.p. 75-7°.

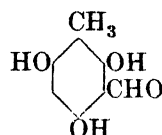
*Me ester*: cryst. from MeOH. M.p. 144-5°.

*Triacetyl*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 163-4°.

Schreier, Wenzel, *Monatsh.*, 1904, **25**, 312.

Herzig, Wenzel, Graetz, *Monatsh.*, 1902, **23**, 100.

**2 : 4 : 6-Trihydroxy-m-toluic Aldehyde**



C<sub>8</sub>H<sub>8</sub>O<sub>4</sub>

MW, 168

Cryst. + ½ H<sub>2</sub>O from H<sub>2</sub>O. Decomp. at 130°. Sol. EtOH, AcOH, AcOEt. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin.

*Oxime*: yellow cryst. Decomp. at 140°.

Herzig, Wenzel, Kerényi, *Monatsh.*, 1903, **24**, 876.

**2 : 3 : 5-Trihydroxy-p-toluic Aldehyde**.

See Thamamol.

**2 : 2' : 2''-Trihydroxytriethylamine** (*Triethanolamine*)

N(CH<sub>2</sub>·CH<sub>2</sub>OH)<sub>3</sub>

C<sub>6</sub>H<sub>15</sub>O<sub>3</sub>N

MW, 149

Oil. B.p. 277-9°/150 mm. D<sub>20</sub> 1.1242. n<sub>D</sub><sup>20</sup> 1.4852. Darkens in air. Non-volatile in steam. Completely miscible with H<sub>2</sub>O, EtOH. Sol. CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

*B,HCl*: cryst. from EtOH. M.p. 177°. Spar. sol. H<sub>2</sub>O, EtOH.

*B,H AuCl<sub>4</sub>*: plates + H<sub>2</sub>O. M.p. anhyd. 77-8°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: cryst. from EtOH. M.p. 118-19°.

*Triacetyl*: oil. B.p. 206-7°/27 mm. Very sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*N-Oxide*: cryst. from EtOH. M.p. 104-105.5°. Very sol. H<sub>2</sub>O, EtOH. Spar. sol. CHCl<sub>3</sub>, Me<sub>2</sub>CO. Insol. AcOEt, ligroin. Reduces hot NH<sub>3</sub>.AgNO<sub>3</sub> but not Fehling's. Sweet taste. *Picrate*: deliquescent cryst. from EtOH-Et<sub>2</sub>O. M.p. 73-4°.

I.G., F.P., 650,574, (*Chem. Abstracts*, 1929, **23**, 3232).

Jones, Burns, *J. Am. Chem. Soc.*, 1925, **47**, 2966.

Wurtz, *Ann.*, 1862, **121**, 227.

**5 : 7 : 5' - Trihydroxy - 6 : 3' : 4' - trimethoxyisoflavone**.

See Iridenin.

**4 : 6 : 3' - Trihydroxy-5 : 2 : 5' - trimethyl-2-hydroxymethyl-diphenyl Ether**.

See Hyposalazinol.

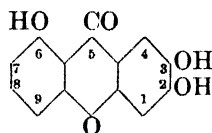
**4 : 4' : 4'' - Trihydroxytriphenylmethane**.

See Leucaurine.

**3 : 6 : 7-Trihydroxytropene**.

See Teloidine.

## 2 : 3 : 6-Trihydroxyxanthone

 $C_{13}H_8O_5$ 

MW, 244

Pale yellow needles +  $2H_2O$  from EtOH. Aq. M.p.  $328-30^\circ$  decomp. Sol. EtOH. Spar. sol.  $H_2O$ . Insol.  $Me_2CO$ , Py. Sol. alkalis with yellow col.

6-Me ether:  $C_{14}H_{10}O_5$ . MW, 258. Needles from EtOH. Decomp. at  $249-51^\circ$ . Diacetyl: needles from EtOH. M.p.  $231-2^\circ$  decomp.

2 : 3-Di-Me ether:  $C_{15}H_{12}O_5$ . MW, 272. Yellow needles from EtOH. M.p.  $228-31^\circ$  decomp. Acetyl: needles from EtOH. M.p.  $224-5^\circ$  decomp.

Tri-Me ether:  $C_{16}H_{14}O_5$ . MW, 286. Yellow needles from EtOH. M.p.  $194-5^\circ$ .

Triacetyl: yellow needles from EtOH. M.p.  $226-7^\circ$ .

Nierenstein, *Ber.*, 1913, **46**, 650.

Dean, Nierenstein, *J. Chem. Soc.*, 1920, 802.

## 2 : 4 : 7-Trihydroxyxanthone.

See Gentisein.

## Tri-indole.

See under Indole.

## Tri-iodoacetanilide.

See under Tri-iodoaniline.

## Tri-iodoacetic Acid

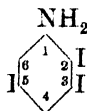
 $CI_3 \cdot COOH$  $C_2HO_2I_3$ 

MW, 438

Yellow leaflets. M.p.  $150^\circ$  decomp. Warm AcOH  $\rightarrow CO_2$  + iodoform.

Angeli, *Ber.*, 1893, **26**, 596.

## 2 : 3 : 5-Tri-iodoaniline

 $C_6H_4NI_3$ 

MW, 471

Needles. M.p.  $116^\circ$ . Mod. sol. EtOH, AcOH,  $C_6H_6$ . Spar. sol.  $Et_2O$ , ligroin.

Acetyl: 2 : 3 : 5-tri-iodoacetanilide. Needles. M.p.  $227^\circ$ . Sublimes slowly above  $200^\circ$ . Spar. sol. usual solvents.

Brenans, *Bull. soc. chim.*, 1904, **31**, 131.

## 2 : 3 : 6-Tri-iodoaniline.

Needles from EtOH or EtOH- $Et_2O$ . M.p.  $116-8^\circ$ .

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 689.

## 2 : 4 : 5-Tri-iodoaniline.

Needles from EtOH- $Et_2O$ . M.p.  $117-8^\circ$ .

Acetyl: 2 : 4 : 5-tri-iodoacetanilide. Needles. M.p.  $241-5^\circ$ . Spar. sol. EtOH,  $Et_2O$ .

See previous reference.

## 2 : 4 : 6-Tri-iodoaniline.

Needles from EtOH. M.p.  $185-5^\circ$  ( $184^\circ$ ). Sol. AcOH,  $CS_2$ . Mod. sol. hot EtOH.

Wheeler, Johns, *Am. Chem. J.*, 1910, **43**, 405.

Michael, Norton, *Ber.*, 1878, **11**, 111.

Jackson, Whitmore, *J. Am. Chem. Soc.*, 1915, **37**, 1528.

## 3 : 4 : 5-Tri-iodoaniline.

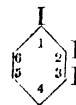
Needles from EtOH- $Me_2CO$ . M.p.  $174-5^\circ$  decomp. Very sol.  $CHCl_3$ ,  $Me_2CO$ , pet. ether. Sol.  $C_6H_6$ . Mod. sol. EtOH,  $Et_2O$ .

Kalb, Schweizer, Zellner, Berthold, *Ber.*, 1926, **59**, 1867.

## Tri-iodoanisole.

See under Tri-iodophenol.

## 1 : 2 : 3-Tri-iodobenzene

 $C_6H_3I_3$ 

MW, 456

Needles from EtOH, prisms from  $C_6H_6$ . M.p.  $116^\circ$ . Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ .

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 687.

See also Kalb, Schweizer, Zellner, Berthold, *Ber.*, 1926, **59**, 1862.

## 1 : 2 : 4-Tri-iodobenzene.

Needles from EtOH. M.p.  $91-5^\circ$  ( $77^\circ$ ). Sol. EtOH,  $CHCl_3$ .

Körner, Belasio, *Atti accad. Lincei*, 1908, **17**, I, 683.

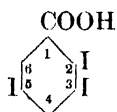
Brenans, *Bull. soc. chim.*, 1914, **15**, 383.

## 1 : 3 : 5-Tri-iodobenzene.

Needles from AcOH. M.p.  $184-2^\circ$  ( $180^\circ$ ). Sol. boiling AcOH. Spar. sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Insol.  $H_2O$ . Volatile in steam.

Jackson, Behr, *Am. Chem. J.*, 1901, **26**, 58.

Körner, Contardi, *Atti accad. Lincei*, 1913, **22**, I, 832.

**2 : 3 : 5-Tri-iodobenzoic Acid**

$C_7H_3O_2I_3$  MW, 500

Prisms from EtOH. M.p. 224–6°. Sol.  $Et_2O$ , hot EtOH. Spar. sol. boiling  $C_6H_6$ . Insol.  $H_2O$ .

Wheeler, Johns, *Am. Chem. J.*, 1910, **43**, 407.

**2 : 4 : 5-Tri-iodobenzoic Acid.**

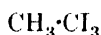
Needles from EtOH. M.p. 248°. Mod. sol. hot EtOH,  $Et_2O$ . Insol. hot  $H_2O$ ,  $C_6H_6$ .

Wheeler, Johns, *Am. Chem. J.*, 1910, **44**, 451.

**3 : 4 : 5-Tri-iodobenzoic Acid.**

Prisms from EtOH. M.p. 288°. Sol. EtOH. Insol.  $H_2O$ .

Wheeler, Liddle, *Am. Chem. J.*, 1909, **42**, 458.

**1 : 1 : 1-Tri-iodoethane (Methyliodoform)**

$C_2H_3I_3$  MW, 408

Yellow cryst. from EtOH. M.p. 95° decomp. Very sol.  $Et_2O$ ,  $C_6H_6$ ,  $CS_2$ . Spar. sol. EtOH, ligroin.

Boissieu, *Bull. soc. chim.*, 1888, **49**, 16.

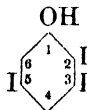
Emschwiller, *Compt. rend.*, 1933, **196**, 1028.

**Tri-iodomethane.**

See Iodoform.

**Tri-iodophenetole.**

See under Tri-iodophenol.

**2 : 3 : 5-Tri-iodophenol**

$C_6H_3OI_3$  MW, 472

Prismatic needles from  $C_6H_6$ -ligroin. M.p. 114°. Sol. usual org. solvents.

*Et ether* : 2 : 3 : 5-tri-iodophenetole.  $C_8H_7OI_3$ . MW, 500. Needles. M.p. 121°.

*Acetyl* : needles. M.p. 123°. Sol. EtOH, AcOH.

Brenans, *Bull. soc. chim.*, 1904, **31**, 132; 1914, **15**, 383.

**2 : 4 : 6-Tri-iodophenol.**

Needles from EtOH.Aq. M.p. 158–9°. Sol. 50 parts 95% EtOH. Mod. sol.  $Et_2O$ ,  $Me_2CO$ . Spar. volatile in steam. Warm conc.  $HNO_3 \rightarrow$  picric acid.

*Me ether* : 2 : 4 : 6-tri-iodoanisole.  $C_7H_5OI_3$ . MW, 486. Leaflets from  $C_6H_6$ , needles from  $Et_2O$ . M.p. 98–9°.

*Et ether* : 2 : 4 : 6-tri-iodophenetole. Prismatic needles from  $Et_2O$ . M.p. 83°.

*Propyl ether* :  $C_9H_9OI_3$ . MW, 514. Needles. M.p. 81°.

*Allyl ether* :  $C_9H_7OI_3$ . MW, 512. Needles from  $C_6H_6$ . M.p. 113–14°. Spar. sol.  $Et_2O$ .

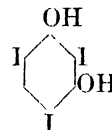
*Acetyl* : needles or prisms from  $C_6H_6$ . M.p. 156°.

*p*-Nitrobenzoyl : m.p. 181°.

Brenans, *Bull. soc. chim.*, 1901, **25**, 630, 820.

Marsh, *J. Chem. Soc.*, 1927, 3164.

Brenans, Girod, *Compt. rend.*, 1928, **186**, 1851.

**2 : 4 : 6-Tri-iodoresorcinol**

$C_6H_3O_2I_3$  MW, 488

Needles from  $CS_2$ . M.p. 145°. Sol. EtOH,  $Et_2O$ ,  $CS_2$ . Spar. sol. hot  $H_2O$ .

*Diacetyl* : needles. M.p. 170°. Sol. EtOH,  $Et_2O$ .

Claassen, *Ber.*, 1878, **11**, 1442.

Michael, Norton, *Ber.*, 1876, **9**, 1752.

**2 : 3 : 4-Tri-iodotoluene**

$C_7H_5I_3$  MW, 470

Light brown needles from EtOH. M.p. 92°. Sol.  $C_6H_6$ . Mod. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, **44**, 506.

**2 : 3 : 5-Tri-iodotoluene.**

Orange plates from EtOH. M.p. 72–3°. Spar. volatile in steam.

See previous reference.

**2 : 3 : 6-Tri-iodotoluene.**

Needles from EtOH. M.p. 80–5°. Spar. sol. EtOH. Spar. volatile in steam.

Wheeler, *Am. Chem. J.*, 1910, **44**, 135.

**2 : 4 : 5-Tri-iodotoluene.**

Brownish needles from EtOH. M.p. 118–20°. Spar. volatile in steam.

Wheeler, *Am. Chem. J.*, 1910, **44**, 140, 500.  
Cf. Neumann, *Ann.*, 1887, **241**, 56.

**2 : 4 : 6-Tri-iodotoluene.**

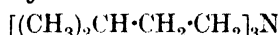
Needles from C<sub>6</sub>H<sub>6</sub>. M.p. 105°. Spar. sol. EtOH.

Wheeler, *Am. Chem. J.*, 1910, **44**, 501.  
Cf. Neumann, *Ann.*, 1887, **241**, 56.

**3 : 4 : 5-Tri-iodotoluene.**

Needles from EtOH. M.p. 122–3°.

Wheeler, Liddle, *Am. Chem. J.*, 1909, **42**, 450.

**Tri-isoamylamine**

C<sub>15</sub>H<sub>33</sub>N MW, 227

Liq. B.p. 237° (257°, 265–70°). D<sub>4</sub><sup>15</sup> 0.7882, D<sub>20</sub><sup>20</sup> 0.7859. Heat of comb. C<sub>6</sub> 2452.1 Cal.

B.HCl: cryst. Sol. EtOH, Et<sub>2</sub>O. Spar. sol. H<sub>2</sub>O.

*Isoamyl iodide*: tetra-isoamylammonium iodide. Prisms. M.p. 83°. D<sub>4</sub><sup>15</sup> 1.0914. Spar. sol. H<sub>2</sub>O.

Wallach, *Ann.*, 1905, **343**, 68.

Plimpton, *J. Chem. Soc.*, 1881, **39**, 332.

Hofmann, *Ann.*, 1851, **79**, 22.

Matter, D.R.P., 301,450, (*Chem. Zentr.*, 1918, I, 53).

**Tri-isobutylamine**

C<sub>12</sub>H<sub>27</sub>N MW, 185

F.p. –24°. B.p. 184–6°. D<sub>4</sub><sup>17.3</sup> 0.7711. n<sub>D</sub><sup>17.3</sup> 1.42519. Non-misc. with H<sub>2</sub>O. Heat of comb. C<sub>6</sub> 1969 Cal.

*Methochloroplatinate*: m.p. 174°.

*Ethochloroplatinate*: m.p. 170°.

*Propylchloroplatinate*: m.p. 168°.

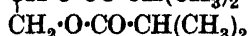
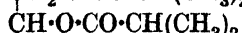
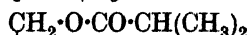
*Butylchloroplatinate*: m.p. 162°.

Ladenburg, *Ber.*, 1879, **12**, 949.

Malbot, *Ann. chim. phys.*, 1888, **13**, 493.

**Tri-isobutyl phosphate.**

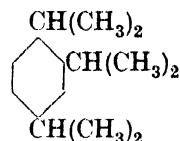
See under Phosphoric Acid.

**Tri-isobutyryn (Glycerol tri-isobutyrate)**

C<sub>15</sub>H<sub>26</sub>O<sub>6</sub> MW, 302

Liq. B.p. 282–4°, 173–6°/24 mm.

Guth, *Zeitschrift für Biologie*, 1903, **44**, 97.

**1 : 2 : 4-Tri-isopropylbenzene**

C<sub>15</sub>H<sub>24</sub>

MW, 204

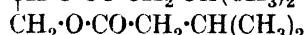
Liq. B.p. 244°, 237°/736 mm., 113–14°/14 mm. D<sub>25</sub><sup>25</sup> 0.8599. n<sub>D</sub><sup>25</sup> 1.4896.

Slanina, Sowa, Nieuwland, *J. Am. Chem. Soc.*, 1935, **57**, 1549.

Kirrmann, Graves, *Bull. soc. chim.*, 1934, **1**, 1494.

**Tri-isopropyl phosphate.**

See under Phosphoric Acid.

**Tri-isovalerin (Isovalerin, glycerol tri-iso-valerate)**

C<sub>18</sub>H<sub>32</sub>O<sub>6</sub>

MW, 344

B.p. 330–5°/763 mm., 209.5–210.5°/27 mm. D<sub>20</sub><sup>20</sup> 0.9984. n<sub>D</sub><sup>20</sup> 1.43535.

Newman, Trikojus, Harker, *Chem. Abstracts*, 1926, **20**, 2658.

**2 : 4 : 6 - Tri keto - 1 : 1 - dimethylcyclo - hexane.**

See Filicinic Acid.

**2 : 4 : 6-Triketo-n-heptane.**

See Diacetylacetone.

**Triketohexane.**

See Hexantrione.

**Triketopentane.**

See Pentantrione.

**1 : 3 : 5-Triketopimelic Acid (Acetone-dioxalic acid, xanthochelidonic acid)**

C<sub>7</sub>H<sub>6</sub>O<sub>7</sub>

MW, 202

Amorph. Very unstable. Sol. H<sub>2</sub>O, EtOH. Less sol. Et<sub>2</sub>O. On standing → chelidonic acid. Forms stable salts.

*Mono K salt*: cryst. ppt. Mod. sol. cold H<sub>2</sub>O.

*Di-Et ester*: C<sub>11</sub>H<sub>14</sub>O<sub>7</sub>. MW, 258. Exists in mono- and di-enol forms. (i) *Mono-enol form*: prisms from EtOH. M.p. 104°. Sol. 60 parts Et<sub>2</sub>O. Mod. sol. hot EtOH, C<sub>6</sub>H<sub>6</sub>. Warm → dienol form. (ii) *Dienol form*: yellow cryst. powder. M.p. 98°. Sol. 40 parts Et<sub>2</sub>O. In

solution is partially converted into monoenol form.

Lerch, *Monatsh.*, 1884, **5**, 375.

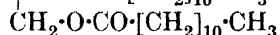
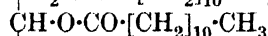
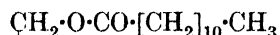
Claisen, *Ber.*, 1891, **24**, 116; D.R.P., 57,648.

Willstätter, Pummerer, *Ber.*, 1904, **37**, 3734.

## 2 : 3 : 4-Triketotetrahydroquinoline.

See Quinisin.

**Trilaurin** (*Glycerol trilaurate, laurin*)



$\text{C}_{39}\text{H}_{74}\text{O}_6$

MW, 638

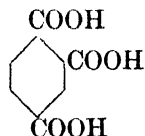
Found in laurel leaves, mahuba seeds and in fats of many other seeds. Needles from EtOH. M.p.  $49^\circ$  ( $45.6^\circ$ ).  $D_{20}^{25}$  0.8944.  $n_D^{20}$  1.44039. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, pet. ether. Spar. sol. cold EtOH. Heat of comb.  $C_v$  4707 Cal.

Bömer, Engel, *Chem. Abstracts*, 1929, **23**, 4676.

André, *Compt. rend.*, 1927, **184**, 227.

Averil, Roche, King, *J. Am. Chem. Soc.*, 1929, **51**, 870.

**Trimellitic Acid** (*Benzene 1 : 2 : 4-tricarboxylic Acid*)



$\text{C}_9\text{H}_6\text{O}_6$

MW, 210

Cryst. from AcOH or EtOH.Aq. M.p.  $238^\circ$  ( $229-30^\circ$ ). Sol. EtOH. Spar. sol. Me<sub>2</sub>CO. Insol. CHCl<sub>3</sub>, CCl<sub>4</sub>, C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>. Sol. 20 parts boiling AcOH.  $k$  (first) =  $3.2 \times 10^{-3}$  at  $25^\circ$ ; (second) =  $1.1 \times 10^{-4}$  at  $25^\circ$ .

1-*Me ester*:  $\text{C}_{10}\text{H}_8\text{O}_6$ . MW, 224. Exists in two forms; m.p.  $177^\circ$ , and m.p.  $203.5-205.5^\circ$ . Sol. H<sub>2</sub>O, Et<sub>2</sub>O, MeOH, hot AcOH (part. decomp.). Spar. sol. CHCl<sub>3</sub>, CCl<sub>4</sub>, CS<sub>2</sub>. Very spar. sol. C<sub>6</sub>H<sub>6</sub>, pet. ether.  $k$  (first) =  $1.8 \times 10^{-3}$  at  $25^\circ$ ; (second) =  $7.8 \times 10^{-5}$  at  $25^\circ$ .

2-*Me ester*: powder. M.p.  $208^\circ$ . Sol. hot H<sub>2</sub>O. Mod. sol. Et<sub>2</sub>O. Insol. CHCl<sub>3</sub>, CCl<sub>4</sub>, CS<sub>2</sub>.  $k$  (first) =  $2.6 \times 10^{-3}$  at  $25^\circ$ ; (second) =  $9.9 \times 10^{-5}$  at  $25^\circ$ .

4-*Me ester*: leaflets from H<sub>2</sub>O. M.p.  $145-7^\circ$ . Sol. Et<sub>2</sub>O. Mod. sol. H<sub>2</sub>O. Insol. pet. ether.  $k$  (first) =  $2.89 \times 10^{-3}$  at  $25^\circ$ ; (second) =  $1.1 \times 10^{-5}$  at  $25^\circ$ .

1 : 2-*Di-Me ester*:  $\text{C}_{11}\text{H}_{10}\text{O}_6$ . MW, 238. Needles from CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub> or Et<sub>2</sub>O-pet. ether. M.p.  $115.5-117^\circ$ , solidifies and then remelts at  $121^\circ$ . B.p. above  $200^\circ/12$  mm.  $k = 3.4 \times 10^{-4}$  at  $25^\circ$ .

*Tri-Me ester*:  $\text{C}_{12}\text{H}_{12}\text{O}_6$ . MW, 252. Thick oil. F.p.  $-13^\circ$ . B.p.  $194^\circ/12$  mm.

*Tri-Et ester*:  $\text{C}_{15}\text{H}_{18}\text{O}_6$ . MW, 294. B.p.  $238^\circ/28$  mm.

1-*Amide*:  $\text{C}_9\text{H}_7\text{O}_5\text{N}$ . MW, 209. Yellowish cryst. from MeOH-C<sub>6</sub>H<sub>6</sub>. M.p.  $185-6^\circ$ . Sol. EtOH. Mod. sol. H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.  $k$  (first) =  $4.4 \times 10^{-4}$  at  $25^\circ$ .

2-*Amide*: cryst. from MeOH-C<sub>6</sub>H<sub>6</sub>. M.p.  $199-200^\circ$ . Sol. EtOH. Mod. sol. H<sub>2</sub>O. Spar. sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.  $k$  (first) =  $7.6 \times 10^{-4}$  at  $25^\circ$ .

4-*Amide*: cryst. from Et<sub>2</sub>O-C<sub>6</sub>H<sub>6</sub>. M.p.  $166^\circ$ . Sol. H<sub>2</sub>O, EtOH. Insol. CHCl<sub>3</sub>, CCl<sub>4</sub>, C<sub>6</sub>H<sub>6</sub>.

2-*Nitrile*:  $\text{C}_9\text{H}_5\text{O}_4\text{N}$ . MW, 191. Yellow amorph. mass. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

*Anhydride*: anhydrotrimellitic acid. Needles. M.p.  $162.5-163.5^\circ$  ( $157^\circ$ ). B.p.  $240-5^\circ/14$  mm. Sol. hot H<sub>2</sub>O. *Me ester*: cryst. M.p.  $94-9^\circ$ .

Schultze, *Ann.*, 1908, **359**, 143.

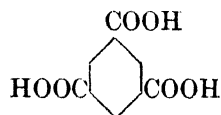
Wegscheider, Perndanner, Auspitzer, *Monatsh.*, 1910, **31**, 1267.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2554.

## Trimercaptobenzene.

See Trithiophloroglucinol.

**Trimesic Acid** (*Benzene-1 : 3 : 5-tricarboxylic acid*)



$\text{C}_9\text{H}_6\text{O}_6$

MW, 210

Needles or prisms from H<sub>2</sub>O. M.p. about  $380^\circ$ . Very sol. EtOH. Sol. Et<sub>2</sub>O. Sol. 40 parts H<sub>2</sub>O at  $23^\circ$ . Heat of comb.  $C_v$  768.5 Cal.

*Mono-Me ester*:  $\text{C}_{10}\text{H}_8\text{O}_6$ . MW, 224. Cryst. + H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd.  $205-8^\circ$ . Sol. EtOH, Et<sub>2</sub>O.

*Tri-Me ester*:  $\text{C}_{12}\text{H}_{12}\text{O}_6$ . MW, 252. Needles. M.p.  $144^\circ$ .

*Tri-Et ester*:  $\text{C}_{15}\text{H}_{18}\text{O}_6$ . MW, 294. Prisms or needles from EtOH. M.p.  $133.5-134.5^\circ$ . Very sol. Et<sub>2</sub>O, MeOH, AcOEt, CHCl<sub>3</sub>, CS<sub>2</sub>. Sol. hot EtOH, AcOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*Tri-isoamyl ester*:  $\text{C}_{24}\text{H}_{36}\text{O}_6$ . MW, 420. Cryst. M.p.  $28-9^\circ$ . B.p.  $278-80^\circ/15$  mm.



*Trihydrazide*: micro-plates from  $H_2O$ . Decomp. above  $300^\circ$ . Spar. sol. EtOH.

Wolff, Heip, *Ann.*, 1899, **305**, 153.

Wislicenus, v. Wrangell, *Ann.*, 1911, **381**, 372.

Ullmann, Uzbachian, *Ber.*, 1903, **36**, 1799.

v. Schaak, U.S.P., 1,706,639, (*Chem. Abstracts*, 1929, **23**, 2187).

### 3 : 4 : 5-Trimethoxy-1-allylbenzene.

See Elemicin.

### 3 : 4 : 5-Trimethoxycinnamic Acid.

See under Sinapic Acid.

### 6 : 7 : 8-Trimethoxycoumarin.

See under Fraxetin.

### 7 : 3' : 5'-Trimethoxydelphinidin chloride.

See Hirsutidin chloride.

### 3 : 4 : 5-Trimethoxyphenylacetic Acid.

See under Iridic Acid.

### 3 : 4 : 5-Trimethoxyphenyl-ethylamine.

See Mescaline.

### 2 : 4 : 5-Trimethoxy-1-propenylbenzene.

See Asarone.

### 3 : 4 : 5-Trimethoxy-1-propenylbenzene.

See under Elemicin.

### Trimethylacetaldehyde.

See Pivalic Aldehyde.

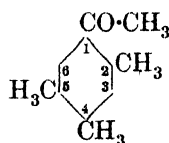
### Trimethylacetic Acid.

See Pivalic Acid.

### 1 : 1 : 1-Trimethylacetone.

See Pinacolin.

### 2 : 4 : 5-Trimethylacetophenone (5-Aceto- $\psi$ -cumene)



$C_{11}H_{14}O$

MW, 162

F.p.  $11^\circ$ . B.p.  $246-7^\circ$ ,  $137-8^\circ/20$  mm.  $D_4^{14.7}$  1.0039.  $n_D^{14.9}$  1.541. Sol. EtOH, Et<sub>2</sub>O, CS<sub>2</sub>, AcOH, C<sub>6</sub>H<sub>6</sub>.

*Oxime*: cryst. from ligroin. M.p.  $85-6^\circ$ .

*Semicarbazone*: plates from EtOH. M.p.  $204^\circ$ .

Auwers, Köckritz, *Ann.*, 1907, **352**, 313.

Klages, Allendorff, *Ber.*, 1898, **31**, 1005.

### 2 : 4 : 6-Trimethylacetophenone (Acetomesitylene).

B.p.  $240.5^\circ/735$  mm.,  $90^\circ/3$  mm.  $D^{20}$  0.9754.  $n_D^{20}$  1.5175.

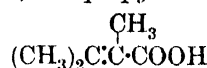
Noller, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1893.

Meyer, Molz, *Ber.*, 1897, **30**, 1271.

### $\omega$ -Trimethylacetophenone.

See *tert*.-Butyl phenyl Ketone.

### Trimethylacrylic Acid (3-Methyl-2-butylene-2-carboxylic acid, 1-isopropylidene-propionic acid)



$C_6H_{10}O_2$

MW, 114

Long needles from  $H_2O$ . M.p.  $70-1^\circ$ . Sol. 20 parts  $H_2O$  at  $19^\circ$ . Sol. hot EtOH, Et<sub>2</sub>O,  $\text{CHCl}_3$ , C<sub>6</sub>H<sub>6</sub>, ligroin. Spar. sol. hot  $H_2O$ .  $k = 3.9 \times 10^{-5}$  at  $25^\circ$ .

*Et ester*:  $C_8H_{14}O_2$ . MW, 142. B.p.  $154-6^\circ$  ( $153-7^\circ/750$  mm.).  $D_4^{19.3}$  0.9072.  $n_D^{19.3}$  1.430.

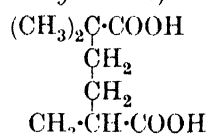
*Nitrile*:  $C_6H_9N$ . MW, 95. B.p.  $155-7^\circ$ .  $D^{18}$  0.8447. Insol.  $H_2O$ .

Perkin, *J. Chem. Soc.*, 1896, **69**, 1479.

Henry, *Chem. Zentr.*, 1899, **I**, 195.

Merling, Welde, *Ann.*, 1909, **366**, 140.

### 1 : 1 : 4-Trimethyladipic Acid (2-Methyl-hexane-2 : 5-dicarboxylic acid)



$C_9H_{16}O_4$

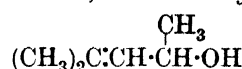
MW, 188

Prisms from  $H_2O$  or dil. formic acid. M.p.  $117^\circ$  ( $114-15^\circ$ ). Mod. sol. dil. formic acid. Spar. sol.  $H_2O$ .

Auwers, Hessenland, *Ber.*, 1908, **41**, 1815.

Wallach, Kempe, *Ann.*, 1903, **329**, 91.

### 1 : 3 : 3-Trimethylallyl Alcohol (1 : 3-Dimethylcrotonyl alcohol, 1 : 3-dimethyl-2-butenol-1)



$C_6H_{12}O$

MW, 100

*d*-.

B.p.  $129^\circ/760$  mm.,  $43^\circ/17$  mm.  $D_4^{20}$  0.8436.  $n_D^{17}$  1.4297.  $[\alpha]_{5461}^{22} + 11.04^\circ$ .

*Acid phthalate*: needles from pet. ether. M.p.  $44^\circ$ .  $[\alpha]_{5461} + 16.7^\circ$  in  $\text{CHCl}_3$ , +  $22.2^\circ$  in C<sub>6</sub>H<sub>6</sub>. *Brucine salt*: needles from Me<sub>2</sub>CO. M.p.  $144^\circ$ .  $[\alpha]_{5461} - 23.0^\circ$  in  $\text{CHCl}_3$ .

*Acetyl*: b.p.  $51^\circ/15$  mm.  $n_D^{20}$  1.4202.  $[\alpha]_{5461}^{22} - 4.72^\circ$ .

*Benzoyl*: b.p.  $139^\circ/17$  mm.  $n_D^{20}$  1.5047.  $[\alpha]_{5461}^{20} + 35.71^\circ$ .

*l*-.

B.p.  $129^\circ/760$  mm.,  $43^\circ/18$  mm.  $n_D^{17}$  1.4298.  $[\alpha]_{5461}^{20} - 2.60^\circ$ .

*Acid phthalate*: needles from pet. ether. M.p.  $44^\circ$ .  $[\alpha]_{5461} - 14.2^\circ$  in CS<sub>2</sub>. *Strychnine*

salt: needles from  $\text{CHCl}_3 + 4$  vols.  $\text{Me}_2\text{CO}$ .  
 $[\alpha]_{5461} - 24.89^\circ$ .

dl.

B.p.  $131-3^\circ$  ( $125-35^\circ$ ).

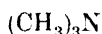
Acid phthalate: prisms from  $\text{Et}_2\text{O}$ -pet. ether.  
 M.p.  $81.5^\circ$ .

Acetyl: b.p.  $50^\circ/15$  mm.  $n_D^{20} 1.4201$ .

Benzoyl: b.p.  $138^\circ/19$  mm.  $n_D^{20} 1.5045$ .

Duveen, Kenyon, *J. Chem. Soc.*, 1936,  
 1451.

### Trimethylamine



$\text{C}_3\text{H}_9\text{N}$

MW, 59

Liq. with fishy odour. M.p.  $-117.2^\circ$ . B.p.  $3.2-3.8^\circ/746.6$  mm.  $D_4^{20} 0.6709$ ,  $D_4^{79} 0.7537$ . Heat of comb.  $C_r$  592.1 Cal.  $k = 7.4 \times 10^{-5}$  at  $25^\circ$ . Crit. temp.  $160.5^\circ$ . Crit. press. 41 atm. Misc. with  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Heat with ethylene chlorohydrin  $\rightarrow$  choline chloride.

$B, \text{H}_2\text{O}$ : m.p.  $5.34^\circ$ .

$B, \text{HCl}$ : needles from  $\text{EtOH}$ . M.p.  $277-8^\circ$  decomp. Sublimes at  $200^\circ$ . Sol.  $\text{EtOH}$ . Mod. sol.  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ .

$B, \text{HBr}$ : prisms from  $\text{EtOH}$ . M.p.  $243-5^\circ$ .

$B, \text{HI}$ : prisms from 95%  $\text{EtOH}$ . M.p.  $263^\circ$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ .

$B, \text{HAuCl}_4$ : yellow prisms from  $\text{EtOH}$ . M.p.  $287^\circ$  ( $237^\circ$ ).

Picrate: pale yellow prisms. M.p.  $216^\circ$ .

Oxide: see Trimethylamine oxide.

Adams, Brown, *Organic Syntheses*, Collective Vol. 1, 514.

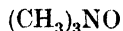
Schmitz, D.R.P., 270,260, (*Chem. Zentr.*, 1914, I, 830).

Koeppen, *Ber.*, 1905, 38, 883.

Schmidt, *Ann.*, 1892, 267, 267.

Dreyfus, E.P., 398,502, (*Chem. Abstracts*, 1934, 28, 1357).

### Trimethylamine oxide (Trimethyloxamine)



$\text{C}_3\text{H}_9\text{ON}$

MW, 75

Widely distributed in fish and animal tissues. Needles  $+ 2\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p.  $255-7^\circ$  ( $208^\circ$ ), anhyd.  $96^\circ$ . Sol.  $\text{H}_2\text{O}$ ,  $\text{MeOH}$ . Less sol.  $\text{EtOH}$ . Heat at  $180^\circ \rightarrow$  formaldehyde + trimethylamine. Reacts strongly alkaline. Does not reduce Fehling's.

$B, \text{HCl}$ : needles from  $\text{EtOH}$ . M.p.  $218^\circ$ . Sol.  $\text{H}_2\text{O}$ , hot  $\text{MeOH}$ .

$B, \text{HI}$ : prisms from  $\text{EtOH}$ . M.p.  $130^\circ$  decomp. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ .

$B, \text{HAuCl}_4$ : yellow cryst. M.p.  $200^\circ$ .

Dict. of Org. Comp.—III.

$B_2, \text{H}_2\text{PtCl}_6$ : m.p.  $228-9^\circ$  decomp.

Picrate: yellow needles. M.p.  $196-8^\circ$ .

Hoppe-Seyler, Schmidt, *Zeitschrift für Biologie*, 1928, 87, 59.

Meisenheimer, *Ann.*, 1913, 397, 287.

Dunstan, Goulding, *J. Chem. Soc.*, 1899, 75, 792, 1005.

### Trimethylamine-tricarboxylic Acid.

See Triglycolamidic Acid.

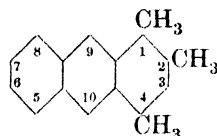
### Trimethyl- $\omega$ -aminoethylcyclopentene.

See Camphylamine.

### 2 : 4 : 6-Trimethylanisole.

See under Mesitol.

### 1 : 2 : 4-Trimethylantracene



$\text{C}_{17}\text{H}_{16}$

MW, 220

Cryst. M.p.  $243^\circ$ .

Gresly, *Ann.*, 1886, 234, 239.

Wende, *Ber.*, 1887, 20, 868.

I.G., E.P., 253,911, (*Chem. Abstracts*, 1927, 21, 2478).

### 1 : 3 : 6-Trimethylantracene.

Cryst. M.p.  $222^\circ$ . Sol.  $\text{Me}_2\text{CO}$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Mod. sol.  $\text{Et}_2\text{O}$ ,  $\text{AcOH}$ . Spar. sol.  $\text{EtOH}$ , ligroin.

Elbs, *J. prakt. Chem.*, 1890, 41, 142.

### 1 : 3 : 10-Trimethylantracene.

Yellow cryst. from pet. ether. M.p.  $100^\circ$ .

Barnett, Hewett, *Ber.*, 1931, 64, 1577.

### 1 : 4 : 9-Trimethylantracene.

Pale yellow cryst. from  $\text{MeOH}$ . M.p.  $81^\circ$ . Sols. show strong fluor.

Barnett, Low, *Ber.*, 1931, 64, 53.

### 2 : 3 : 6-Trimethylantracene.

Found in low-temperature coal tar. Pale yellow flakes with bluish fluor. from boiling  $\text{AcOH}$ . M.p.  $255^\circ$ .

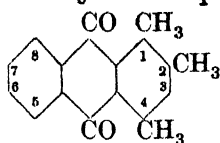
Morgan, Coulson, *J. Chem. Soc.*, 1929, 2555; *J. Soc. Chem. Ind.*, 1934, 53, 71t.

### 2 : 3 : 9-Trimethylantracene.

Pale yellow cryst. from  $\text{AcOEt}$ . M.p.  $125^\circ$ . Sols. show strong fluor.

Barnett, Morrison, *Ber.*, 1931, 64, 538.

## 1 : 2 : 4-Trimethylantraquinone

 $C_{17}H_{14}O_2$ 

MW, 250

Yellow needles from xylene-EtOH. M.p. 162-3°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH.

Gresly, *Ann.*, 1886, **234**, 241.

Elbs, *J. prakt. Chem.*, 1890, **41**, 123.

## 1 : 3 : 6-Trimethylantraquinone.

Pale yellow needles from AcOH. M.p. 192°.

Mayer, Stark, *Ber.*, 1931, **64**, 2010.

Elbs, *J. prakt. Chem.*, 1890, **41**, 143.

## 1 : 3 : 7-Trimethylantraquinone.

Pale yellow needles from AcOH. M.p. 128-9°.

Mayer, Stark, *Ber.*, 1931, **64**, 2010.

## 1 : 4 : 5-Trimethylantraquinone.

Yellow needles from AcOH. M.p. 146-7°.

See previous reference.

## 1 : 4 : 6-Trimethylantraquinone.

Yellow needles from AcOH. M.p. 143°.

See previous reference.

Cf. Elbs, *J. prakt. Chem.*, 1890, **41**, 142.

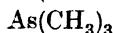
## 2 : 3 : 6-Trimethylantraquinone.

Very pale yellow needles from boiling AcOH. M.p. 240°.

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2555.

Fieser, Seligman, *J. Am. Chem. Soc.*, 1934, **56**, 2695.

## Trimethylarsine (Arsenic trimethyl)

 $C_3H_9As$ 

MW, 120

Colourless liq. B.p. about 70°, (48-51°). Absorbs O from the air to give the oxide. Combines direct with S and halogens. Very poisonous.

$B, HgCl_2$ : needles from hot H<sub>2</sub>O. M.p. 224-6°.

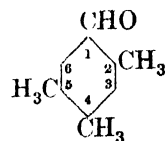
$B, 2HgCl_2$ : plates from hot aq.  $HgCl_2$ . M.p. 264-5° decomp.

Hibbert, *Ber.*, 1906, **39**, 161.

Gryszkiewicz-Trochimowski, Zambrzycki, *Chem. Abstracts*, 1927, **21**, 3612.

Challenger, Higginbottom, Ellis, *J. Chem. Soc.*, 1933, 95.

Challenger, Higginbottom, *Biochem. J.*, 1935, **29**, 1757.

2 : 4 : 5-Trimethylbenzaldehyde (5-Aldehydro-*p*-cumene) $C_{10}H_{12}O$ 

MW, 148

Plates from EtOH. M.p. 43.5° (42°). B.p. 243°, 121°/10 mm. Turns yellow in air.

*Oxime*: needles from ligroin. M.p. 102°.

*Semicarbazone*: prisms from EtOH. M.p. 243-4°.

*Hydrazone*: cryst. M.p. 70°. B.p. 165-6°.

*Picrate*: yellow cryst. powder. M.p. 170-1°.

*Phenylhydrazone*: leaflets from AcOH. M.p. 127°.

Gattermann, *Ann.*, 1906, **347**, 375.

Auwers, Köckritz, *Ann.*, 1907, **352**, 310.

2 : 4 : 6-Trimethylbenzaldehyde (*β*-Isoduryl aldehyde, mesityl aldehyde, aldehydo-mesitylene).

B.p. 237-40°, 192°/50 mm. Fuming HNO<sub>3</sub> + AcOH → 3-nitro-deriv., m.p. 61°.

*Di-Me acetal*: b.p. 242-3°/741 mm.

*Oxime*: *syn*-, needles. M.p. 180-1°. Less sol. than *anti*-form in ord. org. solvents. *Anti*-. Prisms from Et<sub>2</sub>O. M.p. 124°. Heat with dil. HCl → *syn*-form → 2 : 4 : 6-trimethylbenzonitrile. *Acetate*: m.p. 68°.

*Phenylhydrazone*: cryst. from EtOH. Aq. Decomp. in air.

Hinkel, Ayling, Morgan, *J. Chem. Soc.*, 1932, 2797; 1931, 1170.

Wenzel, *Monatsh.*, 1914, **35**, 968.

Fischer, Giebe, *Ber.*, 1898, **31**, 548.

Hantzsch, Lucas, *Ber.*, 1895, **28**, 747.

## 3 : 4 : 5-Trimethylbenzaldehyde (5-Aldehydohemimellitene).

Needles from dil. EtOH. M.p. 52°.

Krömer, *Ber.*, 1891, **24**, 2413.

## 1 : 2 : 3-Trimethylbenzene.

See Hemimellitene.

## 1 : 2 : 4-Trimethylbenzene.

See *p*-Cumene.

## 1 : 3 : 5-Trimethylbenzene.

See Mesitylene.

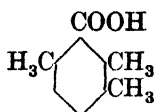
## 2 : 3 : 4-Trimethylbenzoic Acid.

See Prehnitylic Acid.

## 2 : 3 : 5-Trimethylbenzoic Acid.

See *γ*-Isodurylic Acid.

## 2 : 3 : 6-Trimethylbenzoic Acid

 $C_{10}H_{12}O_2$ 

MW, 164

Needles from  $H_2O$  or pet. ether. M.p.  $84^\circ$  (rapid heat.),  $105-6^\circ$  (after solidification).

Lapworth, Wechsler, *J. Chem. Soc.*, 1907, 91, 994.

## 2 : 4 : 5-Trimethylbenzoic Acid.

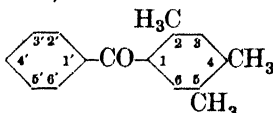
See Durylic Acid.

## 2 : 4 : 6-Trimethylbenzoic Acid.

See  $\beta$ -Isodurylic Acid.

## 3 : 4 : 5-Trimethylbenzoic Acid.

See  $\alpha$ -Isodurylic Acid.

2 : 4 : 5-Trimethylbenzophenone (5-Benzoyl- $\psi$ -cumene) $C_{16}H_{16}O$ 

MW, 224

B.p.  $328^\circ/760$  mm.,  $211^\circ/23$  mm.  $D_4^{25}$  1.0332.

Klages, Allendorff, *Ber.*, 1898, 31, 1001.  
Elbs, *J. prakt. Chem.*, 1887, 35, 491.

## 2 : 4 : 6-Trimethylbenzophenone (Benzoyl-mesitylene).

Prisms from  $EtOH-Et_2O$ . M.p.  $35.5^\circ$ . B.p.  $318-20^\circ$  ( $326.5-327^\circ/777$  mm.),  $189^\circ/17$  mm. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $AcOH$ , pet. ether.

Louise, *Ann. chim. phys.*, 1885, 6, 202.  
Elbs, *J. prakt. Chem.*, 1887, 35, 486.

## 2 : 4 : 4'-Trimethylbenzophenone (4-p-Toluyyl-m-xylene).

Pale yellow viscous oil. B.p.  $340^\circ$ ,  $169^\circ/4$  mm. Does not react with phenylhydrazine or semicarbazide. Reacts only very slowly with hydroxylamine. Hot conc.  $H_2SO_4 \rightarrow p$ -toluic acid + m-xylene.

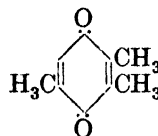
Oxime: cryst. from  $EtOH$ . M.p.  $132^\circ$ .

Morgan, Coulson, *J. Chem. Soc.*, 1929, 2209.

## 2 : 5 : 4'-Trimethylbenzophenone (2-p-Toluyyl-p-xylene).

Rhombic plates from  $EtOH$ . M.p.  $54^\circ$ . B.p.  $337^\circ/760$  mm.,  $202^\circ/23$  mm. Unreactive towards usual ketonic reagents.

See previous reference.

2 : 3 : 5-Trimethyl-p-benzoquinone (2 : 3 : 5-Trimethylquinone,  $\psi$ -cumoquinone) $C_9H_{10}O_2$ 

MW, 150

Yellow needles from  $Et_2O$ . M.p.  $32^\circ$  ( $11^\circ$ ). Volatile in steam.

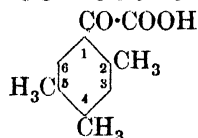
1-Oxime: golden-yellow needles from  $EtOH$ . M.p.  $184^\circ$ . Possesses strong quinone odour.

4-Oxime: yellow needles. M.p.  $134^\circ$ . Odourless.

Noelting, Baumann, *Ber.*, 1885, 18, 1152.

Nietzki, Schneider, *Ber.*, 1894, 27, 1430.

## 2 : 4 : 5-Trimethylbenzoylformic Acid (2 : 4 : 5-Trimethylphenylglyoxylic acid)

 $C_{11}H_{12}O_3$ 

MW, 192

Needles. M.p.  $76^\circ$  ( $61-2^\circ$ ). Sol.  $EtOH$ ,  $Et_2O$ ,  $CHCl_3$ , hot  $H_2O$ .

Et ester:  $C_{13}H_{16}O_3$ . MW, 220. B.p.  $175-6^\circ/10$  mm.

Claus, *J. prakt. Chem.*, 1890, 41, 509.

Bouveault, *Bull. soc. chim.*, 1897, 17, 363, 369.

## 2 : 4 : 6-Trimethylbenzoylformic Acid (2 : 4 : 6-Trimethylphenylglyoxylic acid).

Pale yellow cryst. from  $CS_2$ . M.p.  $117-18^\circ$  ( $114-17^\circ$ ). Sol.  $EtOH$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $CS_2$ , ligroin.  $k = 5.27 \times 10^{-2}$  at  $25^\circ$ . Decomp. on dist.

Me ester:  $C_{12}H_{14}O_3$ . MW, 206. B.p.  $253-5^\circ$  ( $273-5^\circ$ ),  $170^\circ/100$  mm.

Et ester: b.p.  $265-7^\circ$ ,  $164-5^\circ/10$  mm. Turns brown on standing.

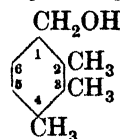
Hydrazone: needles +  $H_2O$ . M.p.  $200^\circ$  decomp. Sol.  $EtOH$ . Mod. sol. boiling  $H_2O$ . Insol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

Bouveault, *Bull. soc. chim.*, 1897, 17, 371.

Claus, *J. prakt. Chem.*, 1890, 41, 504.

Wenzel, *Monatsh.*, 1914, 35, 948.

## 2 : 3 : 4-Trimethylbenzyl Alcohol

 $C_{10}H_{14}O$ 

MW, 150

**2 : 4 : 5-Trimethylbenzyl Alcohol**

Needles from pentane. M.p. 49–50°. B.p. 110–30°/0.5 mm. Sol. usual org. solvents.

Reichstein, Cohen, Ruth, Meldahl, *Helv. Chim. Acta*, 1936, 19, 416.

**2 : 4 : 5-Trimethylbenzyl Alcohol.**

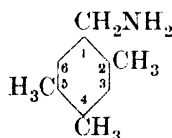
Needles from EtOH. M.p. 168°.

Krömer, *Ber.*, 1891, 24, 2411.

**2 : 4 : 6-Trimethylbenzyl Alcohol (Mesitylcarbinol).**

Needles. M.p. 88–9°. B.p. 140–1°/15 mm. Sol. usual org. solvents.

Carré, *Bull. soc. chim.*, 1910, 7, 842.

**2 : 4 : 5-Trimethylbenzylamine ( $\omega$ -Amino-1 : 2 : 4 : 5-tetramethylbenzene)**

$C_{10}H_{15}N$

MW, 149

Two compounds of this structure have been described in the literature.

(i) Needles from EtOH.Aq. M.p. 64.5°. Sol. EtOH,  $CHCl_3$ . Insol. cold  $H_2O$ .

$B, HCl$ : prisms or needles. M.p. 240–2° decomp.

Krömer, *Ber.*, 1891, 24, 2409.

(ii) Leaflets from MeOH.Aq. M.p. 52°. Sol. usual solvents. Spar. sol. cold  $H_2O$ .

$B, HCl$ : plates. M.p. 275–6°. Sol. hot  $H_2O$ .

*Acetyl*: needles. M.p. 143.5°. Mod. sol.

EtOH,  $C_6H_6$ . Spar. sol.  $H_2O$ .

Willstätter, Kubli, *Ber.*, 1909, 42, 4156.

**3 : 4 : 5-Trimethylbenzylamine ( $\omega$ -Amino-1 : 2 : 3 : 5-tetramethylbenzene).**

Leaflets from boiling  $H_2O$ . M.p. 123°.

$B, HCl$ : needles from EtOH. M.p. 270°.

$B, HAuCl_4$ : red prisms. M.p. 162–5° decomp.

$B_2, H_2PtCl_6$ : rhombohedra. M.p. 219–20° decomp.

*Picrate*: yellow needles from EtOH.Aq. M.p. 239–40° decomp.

Krömer, *Ber.*, 1891, 24, 2411.

**1 : 8 : 8-Trimethylbicyclo-[1, 2, 3]-octanone-2.**

See Homocamphor.

**1 : 8 : 8-Trimethylbicyclo-[1, 2, 3]-octanone-3.**

See Homoepicamphor.

852

**1 : 2 : 2-Trimethyl-3-carboxymethyl-cyclopentane-1-carboxylic Acid****Trimethyl borate (Methyl borate)**

MW, 104

B.p. 55–6° (65°).  $D^0$  0.940,  $D^{20}$  0.915. Burns with green flame. Hyd. by  $H_2O$ .

Schiff, *Ann., Suppl.*, 1867, 5, 183.

Gasselin, *Ann. chim. phys.*, 1894, 3, 22.

Cohn, *Pharmazeutische Zentralhalle*, 1911, 52, 480.

**1 : 1 : 4-Trimethylbutadiene-1 : 3.**

See 2-Methyl-2 : 4-hexadiene.

**1 : 2 : 4-Trimethylbutadiene-1 : 3.**

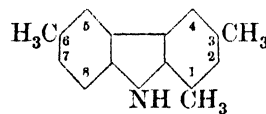
See 3-Methyl-2 : 4-hexadiene.

**1 : 5 : 5-Trimethyl-6- $\alpha$ -butenylcyclohexene.**

See Ionane.

**Trimethyl-*n*-butylmethane.**

See 2 : 2-Dimethyl-*n*-hexane.

**1 : 3 : 6-Trimethylcarbazole**

$C_{15}H_{15}N$

MW, 209

Cryst. from pet. ether. M.p. 126°. Sol. EtOH, AcOH,  $C_6H_6$ .

*N-Nitroso*: yellow needles from pet. ether. M.p. 139°.

Fries, Böker, Wallbaum, *Ann.*, 1934, 509, 94.

**1 : 3 : 7-Trimethylcarbazole.**

Leaflets from EtOH. M.p. 119°.

*Picrate*: deep red cryst. M.p. 177°.

Borsche, *Ann.*, 1908, 359, 77.

**2 : 4 : 6-Trimethylcarbazole.**

Prisms from pet. ether. M.p. 198°.

Fries, Böker, Wallbaum, *Ann.*, 1934, 509, 98.

**Trimethylcarbinol.**

See *tert*.-Butyl Alcohol.

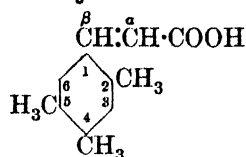
**1 : 2 : 2-Trimethyl-3-carboxycyclopentylacetic Acid.**

See Homoepicamphoric Acid.

**1 : 2 : 2-Trimethyl-3-carboxymethyl-cyclopentane-1-carboxylic Acid.**

See Homocamphoric Acid.

## 2 : 4 : 5-Trimethylcinnamic Acid

 $C_{12}H_{14}O_2$ 

MW, 190

Plates from EtOH.Aq. M.p. 154–5°.

Smith, Tawney, *J. Am. Chem. Soc.*, 1934, **56**, 2169.Smith, Denyes, *J. Am. Chem. Soc.*, 1936, **58**, 306.

## 2 : 4 : 6-Trimethylcinnamic Acid.

Needles from EtOH.Aq. or AcOH.Aq. M.p. 176°. Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>, toluene. Spar. sol. CHCl<sub>3</sub>, CCl<sub>4</sub>, CS<sub>2</sub>.*Et ester*: C<sub>14</sub>H<sub>18</sub>O<sub>2</sub>. MW, 218. Cryst. from EtOH.Aq. or pet. ether. M.p. 40°. B.p. 170°/16 mm. Very sol. EtOH, Et<sub>2</sub>O, AcOH, AcOEt, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin, pet. ether.Böck, Lock, Schmidt, *Monatsh.*, 1934, **64**, 413.

## 2 : 4 : β-Trimethylcinnamic Acid.

*Et ester*: C<sub>14</sub>H<sub>18</sub>O<sub>2</sub>. MW, 218. B.p. 160–70°/20 mm.Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 2540.Mazurewitsch, *Chem. Zentr.*, 1914, I, 1999.

## 2 : 5 : β-Trimethylcinnamic Acid.

*Cis*:

Oil.

*Et ester*: b.p. 150–4°/20 mm., 148–50°/12 mm.*Trans*:

Needles from pet. ether. M.p. 68°. Sol. most solvents.

Auwers, Risse, *Ann.*, 1933, **502**, 294.Ruzicka, Ehmann, Hefti, Altana, *Helv. Chim. Acta*, 1932, **15**, 157.

## 2 : α : β-Trimethylcinnamic Acid.

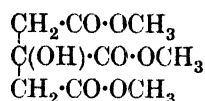
*Et ester*: pale yellow oil. B.p. 128–32°/11 mm.Ruzicka, Ehmann, Hartnagel, Hausschild, *Helv. Chim. Acta*, 1932, **15**, 150.

## 4 : α : β-Trimethylcinnamic Acid.

Leaflets from EtOH.Aq. M.p. 163°.

*Et ester*: b.p. 149–149.5°/10 mm. (141–141.5°/11 mm.).  $D_4^{20}$  1.0024.  $n_D^{20}$  1.519.Auwers, *Ann.*, 1917, **413**, 278.Rupe, Steiger, Fiedler, *Ber.*, 1914, **47**, 73.

## Trimethyl citrate

 $C_9H_{14}O_7$ 

MW, 234

M.p. 78–9°. B.p. 283–7° decomp. → acornitic acid trimethyl ester.

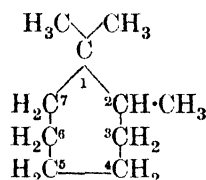
2-Acetyl: 2-acetoxytricarballic trimethyl ester. B.p. 280–2° decomp., 171°/15 mm.

*Me ether*: C<sub>10</sub>H<sub>16</sub>O<sub>7</sub>. MW, 248. B.p. 159–60°/12 mm. Sol. EtOH, Et<sub>2</sub>O.Hunäus, *Ber.*, 1876, **9**, 1750.Anschütz, *Ann.*, 1903, **327**, 229.Saint-Evre, *Ann.*, 1847, **60**, 325.

## Trimethylcycloheptadienone.

See Eucarvone.

## 1 : 1 : 2-Trimethylcycloheptane

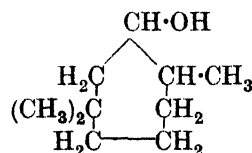
 $C_{10}H_{20}$ 

MW, 140

Liq. B.p. 104–5°/100 mm.  $D_4^{20}$  0.8243.  $n_D^{20}$  1.4527.Ruzicka, Seidel, *Helv. Chim. Acta*, 1936, **19**, 430.1 : 1 : 4-Trimethylcycloheptane (*Eucarvane*).Liq. B.p. 162–3°/720 mm.  $D_4^{20}$  0.8011.  $n_D^{20}$  1.4420.

See previous reference.

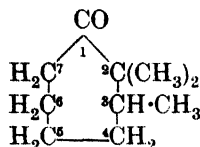
## 2 : 6 : 6-Trimethylcycloheptanol

 $C_{10}H_{20}O$ 

MW, 156

Liq. B.p. 216°.  $D_4^{20}$  0.9096.  $n_D^{20}$  1.4639.*Phenylurethane*: m.p. 76°.Wallach, *Ann.*, 1914, **403**, 90.Wallach, Köhler, *Ann.*, 1905, **339**, 96, 106.

## 2 : 2 : 3-Trimethylcycloheptanone

 $C_{10}H_{18}O$ 

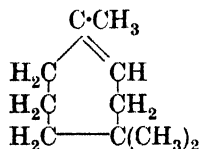
MW, 154

B.p. 80–3°/12 mm.

Semicarbazone : m.p. 169–70°.

Naef, D.R.P., 580,713, (*Chem. Abstracts*, 1934, **28**, 1716).Ruzicka, Seidel, *Helv. Chim. Acta*, 1936, **19**, 430.2 : 6 : 6-Trimethylcycloheptanone (*Tetrahydroeucarvone*).Liq. with peppermint odour. B.p. 208–9°, 85·5°/12 mm.  $D_4^{20}$  0·9095.  $n_D^{18}$  1·4568.

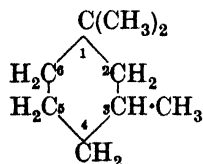
Semicarbazone : exists in two forms. (i) Cryst. M.p. 191–2°. Spar. sol. AcOEt. (ii) Needles. M.p. 161–3°. Sol. AcOEt.

Wallach, *Ann.*, 1911, **381**, 67; 1914, **403**, 89.Wallach, Köhler, *Ann.*, 1905, **339**, 107.1 : 4 : 4-Trimethylcycloheptene (*Eucarvene*) $C_{10}H_{18}$ 

MW, 138

Liq. B.p. 161–5°/720 mm.  $D_4^{20}$  0·8185.  $n_D^{20}$  1·4561.Ruzicka, Seidel, *Helv. Chim. Acta*, 1936, **19**, 431.

## 1 : 1 : 3-Trimethylcyclohexane

 $C_9H_{18}$ 

MW, 126

B.p. 137–8°.  $D_4^{25}$  0·7868.  $n_D^{20}$  1·4362. Heat of comb.  $C_7$  1406 Cal.Auwers, *Ann.*, 1920, **420**, 109.Knoevenagel, *Ann.*, 1897, **297**, 202.1 : 2 : 3-Trimethylcyclohexane (*Hexahydrohemimellitene*).

Cis :

B.p. 144–6°/755 mm.  $D_4^{20}$  0·7930.  $n_D^{20}$  1·43682.

Trans :

B.p. 142–143·5°/762 mm.  $D_4^{20}$  0·7914.  $n_D^{20}$  1·43582.Eisenlohr, *Fortschritte der Chemie, Physik, und Physikalischen Chemie*, 1925, **18**, 552.1 : 2 : 4-Trimethylcyclohexane (*Hexahydro-ψ-cumene*).

Found in petroleum from Maki, Echigo Province.

Cis :

B.p. 146° (142°).  $D_4^{20}$  0·786.  $n_D^{20}$  1·43209.

Trans :

B.p. 140–1°, 138·5–139·5°/755 mm.  $D_4^{20}$  0·7813.  $n_D^{20}$  1·43121.Iimori, Kikuchi, *Chem. Abstracts*, 1928, **22**, 496.Eisenlohr, *Fortschritte der Chemie, Physik, und Physikalischen Chemie*, 1925, **18**, 553.1 : 3 : 5-Trimethylcyclohexane (*Hexahydro-mesitylene*).

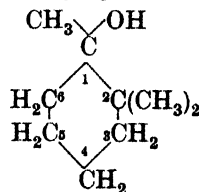
Cis :

B.p. 140·0–140·5°/752 mm.  $D_4^{20}$  0·7773.  $n_D^{20}$  1·43010.

Trans :

B.p. 138·5–139·0°/754 mm.  $D_4^{20}$  0·7720.  $n_D^{20}$  1·42740.Adams, Marshall, *J. Am. Chem. Soc.*, 1928, **50**, 1970.Eisenlohr, *Fortschritte der Chemie, Physik, und Physikalischen Chemie*, 1925, **18**, 554.

## 1 : 2 : 2-Trimethylcyclohexanol

 $C_9H_{18}O$ 

MW, 142

Oil. B.p. 81·4–81·8°/20 mm., 75·8°/16 mm.  $D_4^{18·4}$  0·9274.  $n_D^{18·4}$  1·469.Hydrate,  $\frac{1}{2}H_2O$  : cryst. M.p. 41°.Auwers, Lange, *Ann.*, 1913, **401**, 322.

**1 : 2 : 6-Trimethylcyclohexanol.**B.p. 78°/23 mm.  $D_4^{15}$  0.9126.  $n_D^{15}$  1.4598.Zelinsky, Dvorjantchik, *Bull. soc. chim.*, 1904, **32**, 746.**1 : 3 : 3-Trimethylcyclohexanol.**

Prisms. M.p. 74° (72.5°). Sol. most org. solvents.

Crossley, Gilling, *J. Chem. Soc.*, 1910, **97**, 2220.**1 : 3 : 5-Trimethylcyclohexanol.**B.p. 181°, 82–3°/19 mm.  $D_4^{16.8}$  0.8876.  $n_D^{16.3}$  1.454.Wallach, *Ann.*, 1913, **396**, 284.**1 : 4 : 4-Trimethylcyclohexanol.**

Needles. M.p. 58°. B.p. 79–80°/15 mm., 75°/11 mm.

Auwers, Lange, *Ann.*, 1913, **401**, 317.**2 : 2 : 3-Trimethylcyclohexanol.**

B.p. 85–7°/15 mm.

Ruzicka, *Helv. Chim. Acta*, 1919, **2**, 159.**2 : 2 : 5-Trimethylcyclohexanol.**

See Pulenol.

**2 : 2 : 6-Trimethylcyclohexanol.**Cryst. from EtOH or pet. ether. M.p. 51°. B.p. 186–7°/753 mm., 87°/28 mm.  $D_4^{20}$  0.9128.  $n_D^{20}$  1.4600.Haller, *Compt. rend.*, 1913, **157**, 181.Masson, *Compt. rend.*, 1912, **154**, 518.**2 : 3 : 3-Trimethylcyclohexanol.**

Needles. M.p. 28°. B.p. 197°. Very sol. org. solvents.

Crossley, Renouf, *J. Chem. Soc.*, 1911, **99**, 1108; 1915, **107**, 607.**2 : 3 : 6-Trimethylcyclohexanol.**B.p. 193–5°/747 mm.  $D_4^{17}$  0.9119.Zelinsky, Reformatski, *Ber.*, 1895, **28**, 2945.**2 : 4 : 4-Trimethylcyclohexanol.**

Viscous oil. B.p. 192–3°.

Wallach, Scheunert, *Ann.*, 1902, **324**, 106.**2 : 4 : 5-Trimethylcyclohexanol.***Cis* :

B.p. 191–3°, 84°/17 mm.

*Phenylurethane* : cryst. from EtOH. M.p. 83–5°.*Trans* :

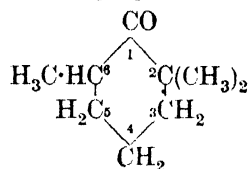
B.p. 196°, 112°/35 mm.

*Phenylurethane* : cryst. from EtOH. M.p. 95°.*Acid phthalate* : cryst. from Et<sub>2</sub>O–ligroin. M.p. 81–83.5°.Skita, *Ber.*, 1920, **53**, 1800.**3 : 3 : 5-Trimethylcyclohexanol (Dihydroisophorol).***Cis* :B.p. 201–3°/750 mm., 92°/12 mm.  $D_4^{16}$  0.9006.  $n_D^{16}$  1.4550.*Acetyl* : b.p. about 209–10°.*Trans* :

Cryst. M.p. 52° (37°). B.p. 196.5°/770 mm., 95°/15 mm.

*Acetyl* : b.p. 209–10°.Skita, Meyer, *Ber.*, 1912, **45**, 3593.Knoevenagel, Fischer, *Ann.*, 1897, **297**, 128, 194.**2 : 2 : 5-Trimethylcyclohexanone.**

See Pulenone.

**2 : 2 : 6-Trimethylcyclohexanone** $C_9H_{16}O$ 

MW, 140

B.p. 178–9°, 66–7°/10 mm.  $D_4^{18}$  0.9043.  $n_D^{19}$  1.4493.*Oxime* : cryst. from EtOH. M.p. 106° (104.5–105°). B.p. 126–7°/17 mm.*Semicarbazone* : m.p. 220–1°.Masson, *Compt. rend.*, 1912, **154**, 517.Haller, Cornubert, *Bull. soc. chim.*, 1927, **41**, 377.Cornubert, *ibid.*, 894.**2 : 3 : 3-Trimethylcyclohexanone.**Liq. B.p. 190.5–191°/750 mm.  $D_{15}^{15}$  0.9213.*Oxime* : needles from pet. ether or EtOH. Aq. M.p. 95°. Sol. most org. solvents.*Semicarbazone* : cryst. from EtOH. M.p. 206° decomp. Spar. sol. AcOEt, CHCl<sub>3</sub>, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>.Crossley, Renouf, *J. Chem. Soc.*, 1911, **99**, 1110.**2 : 3 : 6-Trimethylcyclohexanone.**B.p. 190–1°, 79–80°/20 mm.  $D_4^{18}$  0.9129,  $D_4^{21}$  0.9058.  $n_D^{21}$  1.4464. Non-misc. with H<sub>2</sub>O. Does not form bisulphite comp.Cornubert, Humeau, *Bull. soc. chim.*, 1931, **49**, 1483.Zelinsky, Reformatski, *Ber.*, 1895, **28**, 2944.



**2 : 4 : 4-Trimethylcyclohexanone.**

Present in acetone oil. B.p. 191°, 61°/11 mm.  $D_4^{20}$  0.902.  $n_D^{20}$  1.4493.

*Oxime*: cryst. from  $Et_2O$ . M.p. 108-9°.

*Semicarbazone*: m.p. 164-5° (162°).

Auwers, *Ann.*, 1920, **420**, 111.

Pringsheim, Bondi, *Ber.*, 1925, **58**, 1414.

Wallach, Scheunert, *Ann.*, 1902, **324**, 107.

**2 : 4 : 5-Trimethylcyclohexanone.**

*Cis*:

B.p. 193°.  $D_4^{20}$  0.897.  $n_D^{20}$  1.4479.

*Oxime*: cryst. from MeOH. M.p. 105°.

*Semicarbazone*: needles from MeOH. M.p. 204° (175-5-176°).

Skita, *Ber.*, 1920, **53**, 1800.

Auwers, *Ann.*, 1920, **420**, 103.

**2 : 4 : 6-Trimethylcyclohexanone.**

B.p. 184-5°/748 mm.  $D_4^{20}$  0.8992.  $n_D^{20}$  1.4458.

*Semicarbazone*: m.p. 228°.

Cornubert, Maurel, *Bull. soc. chim.*, 1931, **49**, 1528.

Haller, *Compt. rend.*, 1913, **157**, 740.

**2 : 5 : 5-Trimethylcyclohexanone.**

B.p. 185°.

*Semicarbazone*: m.p. 170°.

Blanc, *Bull. soc. chim.*, 1908, **3**, 786.

**3 : 3 : 4-Trimethylcyclohexanone.**

B.p. 184-8°, 70-5°/11 mm.

*Oxime*: cryst. from  $H_2O$ . M.p. 100°.

*Semicarbazone*: m.p. 177°.

v. Braun, Keller, Weissbach, *Ann.*, 1931, **490**, 188.

**3 : 3 : 5-Trimethylcyclohexanone (Dihydroisophorone).**

Present in acetone oil. Yellow oil. B.p. 188.5-189.5°, 53.5-54°/11 mm.  $D_4^{19}$  0.8919.  $n_D^{15}$  1.4454.

*Oxime*: cryst. from EtOH. M.p. 58°.

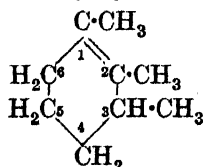
*Semicarbazone*: cryst. from MeOH. M.p. 204° (202°).

2 : 4-Dinitrophenylhydrazone: m.p. 145-7°.

Pringsheim, Bondi, *Ber.*, 1925, **58**, 1414.

Knoevenagel, *Ann.*, 1897, **297**, 198.

Skita, *Ber.*, 1909, **42**, 1630.

**1 : 2 : 3-Trimethylcyclohexene**

$C_9H_{16}$

MW, 124

B.p. 149.6-150°/749 mm.  $D_4^{11.75}$  0.8347.  $n_D^{11.75}$  1.463.

Auwers, Krollpfeiffer, *Ber.*, 1915, **48**, 1231.

**1 : 3 : 5-Trimethylcyclohexene (Tetrahydro-mesitylene).**

B.p. 142.5-143.5°.  $D_4^{14.5}$  0.8025.  $n_D^{13.5}$  1.449.

*Nitroschloride*: m.p. 134°.

Wallach, *Ann.*, 1913, **396**, 284.

Auwers, Hinterseber, Treppmann, *Ann.*, 1915, **410**, 270.

**1 : 4 : 4-Trimethylcyclohexene.**

See Pulecene.

**1 : 4 : 5-Trimethylcyclohexene (1 : 2 : 3 : 6-Tetrahydro-ψ-cumene).**

B.p. 144-6°.  $D_4^{20}$  0.805.  $n_D^{20}$  1.4482.

Auwers, *Ann.*, 1920, **420**, 105.

**1 : 5 : 5-Trimethylcyclohexene (α-Cyclo-geraniolene).**

B.p. 139-41°/759 mm.  $D_4^{23}$  0.7981.  $n_D^{21.5}$  1.44612.

*Nitrosate*: cryst. M.p. 102-4°.

*Nitroschloride*: bluish cryst. from MeOH.Aq. M.p. 100-20°.

Wallach, Scheunert, *Ann.*, 1902, **324**, 101.

Crossley, Gilling, *J. Chem. Soc.*, 1910, **97**, 2221.

**1 : 6 : 6-Trimethylcyclohexene.**

Liq. B.p. 146.2-147.2°/767 mm. (144-6°).  $D_4^{20.3}$  0.8217.  $n_D^{20.4}$  1.456.

*Nitroschloride*: needles from AcOEt. M.p. 133-4°. Insol. MeOH,  $Me_2CO$ .

Auwers, Lange, *Ann.*, 1915, **409**, 174.

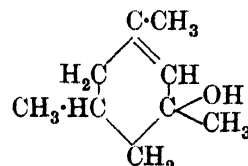
Godchot, Bedos, *Compt. rend.*, 1925, **181**, 921.

**1 : 1 : 3-Trimethylcyclohexene - carb - oxylic Acid.**

See Cyclogeranic Acid.

**1 : 2 : 2-Trimethylcyclohexene-3-carb - oxylic Acid-1.**

See Camphorenic Acid.

**1 : 3 : 5-Trimethylcyclohexenol-3**

$C_9H_{16}O$

MW, 140

Cryst. M.p. 46°. B.p. 87-90°/17 mm.  $D_4^{20.2}$  0.9132.  $n_D^{19.3}$  1.4735. Heat of comb.  $C_9$  1350-9 Cal.

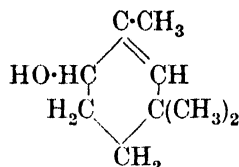
Auwers, Peters, *Ber.*, 1910, **43**, 3087.

### 3 : 3 : 6-Trimethylcyclohexenol-4

#### 3 : 3 : 6-Trimethylcyclohexenol-4.

See  $\beta\gamma$ -Pulnenol.

#### 1 : 3 : 3-Trimethylcyclohexenol-6



$C_9H_{16}O$

MW, 140

Oil. B.p.  $193^\circ/760$  mm.  $D_4^{13}$  0.9310. Misc. with usual org. solvents. Insol.  $H_2O$ . Volatile in steam.

Acetyl: b.p.  $206-7^\circ$ .

Bougault, *Compt. rend.*, 1910, **150**, 534.

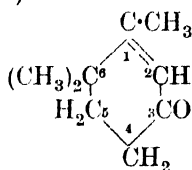
#### 1 : 4 : 4-Trimethylcyclohexenone-3.

See  $\alpha\beta$ -Pulnenone.

#### 1 : 5 : 5-Trimethylcyclohexenone-3.

See Isophorone.

#### 1 : 6 : 6-Trimethylcyclohexenone-3 (Isocamphorophorone)



$C_9H_{14}O$

MW, 138

Liq. B.p.  $217^\circ/760$  mm.,  $97-9^\circ/13$  mm.  $D_4^{20}$  0.9424.  $n_D^{20}$  1.48458.

Semicarbazone: needles from AcOEt. M.p.  $211^\circ$ .

Tiemann, *Ber.*, 1897, **30**, 249.

#### 2 : 6 : 6-Trimethylcyclohexenone-3.

B.p.  $194-6^\circ$  ( $192^\circ$ ).  $D_4^{20}$  0.930.  $n_D^{20}$  1.4779.

Oxime: prisms from  $Et_2O$ -ligroin. M.p.  $128-9^\circ$ . B.p.  $131-2^\circ/15$  mm.

Semicarbazone: cryst. from MeOH.Aq. M.p.  $158-9^\circ$ . Mod. sol.  $Et_2O$ .

Bougault, *Compt. rend.*, 1910, **150**, 534.

Auwers, *Ann.*, 1920, **420**, 110.

Wallach, Scheunert, *Ann.*, 1902, **324**, 103.

#### 4 : 4 : 5-Trimethylcyclohexenone-3.

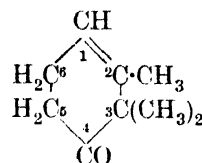
B.p.  $85-90^\circ/14$  mm.

Semicarbazone: cryst. from EtOH. M.p.  $185-7^\circ$ .

Ruzicka, *Helv. Chim. Acta*, 1919, **2**, 159.

### 857 1 : 2 : 3-Trimethylcyclopentane-1-carboxylic Acid

#### 2 : 3 : 3-Trimethylcyclohexenone-4



$C_9H_{14}O$

MW, 138

B.p.  $85-90^\circ/12$  mm.

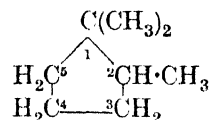
Semicarbazone: cryst. from MeOH. M.p.  $168-71^\circ$ .

Ruzicka, *Helv. Chim. Acta*, 1919, **2**, 158.

#### 3 : 3 : 6-Trimethylcyclohexenone-4.

See  $\beta\gamma$ -Pulnenone.

#### 1 : 1 : 2-Trimethylcyclopentane (Camphorceane, dihydroisolauroleone)



$C_8H_{16}$

MW, 112

Liq. with camphoraceous odour. B.p.  $113-113.5^\circ/750$  mm.  $D_4^{18}$  0.7728,  $D_4^{20}$  0.7661.  $n_D^{18}$  1.4238,  $n_D^{20}$  1.4199. Heat of comb.  $C_v$  1252.8 Cal. Dil.  $HNO_3 \rightarrow$  1 : 1-dimethylglutaric acid.

Zelinsky, Lepeschkin, *Ann.*, 1901, **319**, 315.

Crossley, Renouf, *J. Chem. Soc.*, 1906, **89**, 43.

Kishner, *Chem. Zentr.*, 1911, **I**, 543.

#### 1 : 1 : 3-Trimethylcyclopentane.

B.p.  $115-16^\circ/760$  mm.  $D_4^{20}$  0.7703.  $n_D^{20}$  1.4223.

Zelinsky, Uspensky, *Ber.*, 1913, **46**, 1470.

Dey, Linstead, *J. Chem. Soc.*, 1935, 1064.

#### 1 : 2 : 3-Trimethylcyclopentane (Dihydro-lauroleone, laurolan).

B.p.  $114-15^\circ$ .  $D_4^{20}$  0.7718,  $D_4^{19}$  0.7688.  $n_D^{19}$  1.4230.  $HNO_3 \rightarrow$  oxalic acid.

Zelinsky, Lepeschkin, *Chem. Zentr.*, 1902, **I**, 33.

Crossley, Renouf, *J. Chem. Soc.*, 1906, **89**, 27, 40.

#### 1 : 2 : 4-Trimethylcyclopentane.

B.p.  $112.5-113^\circ$ .  $D_4^{20}$  0.7565.  $n_D^{20}$  1.4156. Heat of comb.  $C_v$  1255.7 Cal.

Zelinsky, *Bull. soc. chim.*, 1904, **32**, 747.

#### 1 : 2 : 3-Trimethylcyclopentane-1-carboxylic Acid.

See Laurolanic Acid.

**2 : 2 : 3-Trimethylcyclopentane-1-carboxylic Acid** 858

**2 : 2 : 3-Trimethylcyclopentane-1-carboxylic Acid.**

See Norisocampholic Acid.

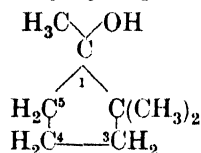
**1 : 1 : 2-Trimethylcyclopentane-5-carboxylic Acid.**

See Lauronic Acid.

**1 : 2 : 2-Trimethylcyclopentane-1 : 3-dicarboxylic Acid.**

See Camphoric Acid.

**1 : 2 : 2-Trimethylcyclopentanol**



$C_8H_{16}O$

MW, 128

Liq. B.p.  $156^\circ/755$  mm.,  $80-1^\circ/49$  mm.,  $60^\circ/15$  mm.  $D_4^{20}$  0.9102.  $n_D^{20}$  1.4513. Dist. with cryst. oxalic acid  $\rightarrow$  isolaurolene.

Hydrate,  $\frac{1}{2}H_2O$ : needles from  $Et_2O$ . M.p.  $59-60^\circ$ .

Kishner, *Chem. Zentr.*, 1911, I, 543.

**1 : 2 : 4-Trimethylcyclopentanol.**

d.-

B.p.  $157-8^\circ/747$  mm.  $D_4^{21}$  0.8850.  $n_D^{21}$  1.4424.  $[\alpha]_D + 15^\circ$ .

Zelinsky, *Bull. soc. chim.*, 1904, 32, 747.

**1 : 2 : 5-Trimethylcyclopentanol.**

B.p.  $56-60^\circ/8$  mm.  $D_4^{15}$  0.9121.  $n_D^{15.7}$  1.4554. Heat with anhyd. oxalic acid or  $P_2O_5 \rightarrow$  laurolene.

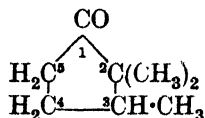
Noyes, Kyriakides, *J. Am. Chem. Soc.*, 1910, 32, 1065.

**2 : 3 : 4-Trimethylcyclopentanol.**

Liq. with menthol odour. B.p.  $68-70^\circ/12$  mm. Insol.  $H_2O$ .

Willstätter, Clarke, *Ber.*, 1914, 47, 309.

**2 : 2 : 3-Trimethylcyclopentanone**



$C_8H_{14}O$

MW, 126

Exists in active and inactive forms.

Active form :

Liq. B.p.  $164-5^\circ$ .

Oxime : leaflets from pet. ether. M.p.  $107-8^\circ$ . Sol.  $EtOH$ , pet. ether.

Semicarbazone : m.p.  $188^\circ$ . Spar. sol. cold  $EtOH$ .

**3 : 3 : 5-Trimethylcyclopentanone**

Inactive form :

B.p.  $164^\circ$ .

Oxime : plates. M.p.  $105^\circ$ .

Semicarbazone : m.p.  $210-12^\circ$ .

Blaise, Blanc, *Bull. soc. chim.*, 1902, 27, 76.

Blanc, Desfontaines, *Compt. rend.*, 1903, 136, 1143.

**2 : 2 : 5-Trimethylcyclopentanone.**

Exists in active and inactive forms.

Active form :

B.p.  $152-3^\circ$ .

Oxime : cryst. from  $MeOH.Aq$ . M.p.  $60-2^\circ$ .

Very sol. org. solvents.

Semicarbazone : m.p.  $150-1^\circ$ . Very sol.  $MeOH$ ,  $Et_2O$ .

Inactive form :

B.p.  $152^\circ$ .  $D_4^{20}$  0.8781.  $n_D^{20}$  1.4306.

Oxime : prisms from pet. ether. M.p.  $62^\circ$ .

Blanc, *Bull. soc. chim.*, 1908, 3, 290, 782.

Haller, Cornubert, *Compt. rend.*, 1914, 158, 300.

Wallach, Kempe, *Ann.*, 1903, 329, 94.

**2 : 3 : 3-Trimethylcyclopentanone.**

Liq. B.p.  $167-9^\circ/760$  mm.  $D_4^{20}$  0.8956. Insol.  $H_2O$ .

Oxime : needles from  $EtOH.Aq$ . M.p.  $105.5^\circ$ .

Semicarbazone : plates from  $EtOH$ . M.p.  $221.5-222^\circ$  decomp.

van Kregten, *Rec. trav. chim.*, 1916, 36, 77.

Noyes, *Ber.*, 1899, 32, 2291.

**2 : 3 : 5-Trimethylcyclopentanone.**

d.-

B.p.  $158-9^\circ/770$  mm.  $D_4^{19}$  0.8778.  $n_D^{19}$  1.4316.  $[\alpha]_D^{19} + 103^\circ 41'$ .

Haller, Cornubert, *Compt. rend.*, 1914, 158, 1619.

**3 : 3 : 4-Trimethylcyclopentanone.**

Liq. B.p.  $172-4^\circ$ .  $n_D^{20}$  1.4390.

Oxime : liq. B.p.  $116-20^\circ/14$  mm.

Semicarbazone : cryst. from  $MeOH$ . M.p.  $162-3^\circ$ .

v. Braun, *Ann.*, 1931, 490, 132.

See also v. Braun, Mannes, Reuter, *Ber.*, 1933, 66, 1499.

**3 : 3 : 5-Trimethylcyclopentanone.**

B.p.  $160-1^\circ$ .  $D_4^{18}$  0.8785.  $n_D^{18}$  1.433.

Semicarbazone : cryst. from  $EtOH$ . M.p.  $171-3^\circ$ .

Dey, Linstead, *J. Chem. Soc.*, 1935, 1063.

Wallach, *Ann.*, 1918, 414, 331.

**1 : 2 : 3-Trimethylcyclopentene.**

See Laurolene.

**1 : 5 : 5-Trimethylcyclopentene.**

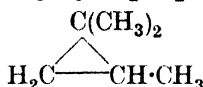
See Isolaurole.

**1 : 1 : 2-Trimethylcyclopentene-2-carboxylic Acid-3.**See  $\beta$ -Campholytic Acid.**1 : 1 : 2-Trimethylcyclopentene-2-carboxylic Acid-5.**See  $\alpha$ -Campholytic Acid.**1 : 2 : 3-Trimethylcyclopentene-3-carboxylic Acid.**

See Laurolenic Acid.

**2 : 2 : 3-Trimethylcyclopentylacetic Acid.**

See Isocampholic Acid.

**1 : 1 : 2-Trimethylcyclopropane** $\text{C}_6\text{H}_{12}$ 

MW, 84

B.p.  $56-7^\circ/750$  mm. ( $52.8^\circ/756$  mm.).  $D_4^{15.3}$  0.6888,  $D_4^{19.5}$  0.6822.  $n_D^{14.5}$  1.3896,  $n_D^{19.5}$  1.3848. Polymerises on shaking with conc.  $\text{H}_2\text{SO}_4$ .

Kishner, *Chem. Zentr.*, 1912, I, 2025.Zelinsky, Zelikow, *Ber.*, 1901, **34**, 2859.**1 : 2 : 3-Trimethylcyclopropane.**B.p.  $65-7^\circ/755$  mm.  $D_4^{18}$  0.6946.  $n_D^{18}$  1.3945.Zelinsky, Zelikow, *Ber.*, 1901, **34**, 2863.**1 : 3 : 5-Trimethyldiphenylmethane.**

See Phenylmesityl methane.

**Trimethyldiphenyl sulphide.**

See under Thioxylene.

**2 : 6 : 10-Trimethyldodecane.**

See Farnesane.

**Trimethylene.**

See Cyclopropane.

**Trimethylene-aniline.**

See under Trimethyleneimine.

**Trimethylene bromide.**

See 1 : 3-Dibromopropane.

**Trimethylene bromohydrin.**

See 3-Bromopropyl Alcohol.

**Trimethylene chloride.**

See 1 : 3-Dichloropropane.

**Trimethylene chlorobromide.**

See 1-Chloro-3-bromopropane.

**Trimethylene chlorohydrin.**

See 3-Chloropropyl Alcohol.

**Trimethylene cyanide.**

See under Glutaric Acid.

**Trimethylenediamine (1 : 3-Diaminopropane)** $\text{C}_3\text{H}_{10}\text{N}_2$ 

MW, 74

Liq. with ammoniacal odour. B.p.  $135-6^\circ/738$  mm.  $D_4^{25}$  0.884. Misc. with  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .  $k = 3.5 \times 10^{-4}$  at  $25^\circ$ .

$B_2\text{HCl}$ : cryst. M.p.  $243^\circ$ . Sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ .

$B_2\text{H}_2\text{PtCl}_6$ : m.p.  $240^\circ$  decomp.

1 : 3-N-Tetra-Me :  $\text{C}_7\text{H}_{18}\text{N}_2$ . MW, 130. Oil. B.p.  $144^\circ/760$  mm.  $D_4^{18.7}$  0.7837.  $n_D^{18.7}$  1.4215. Misc. with  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .  $B_2\text{H}_2\text{PtCl}_6$ : cryst. from 50%  $\text{EtOH}$ . Decomp. at  $246-7^\circ$ . Picrate: cryst. M.p.  $205^\circ$ .

1-N-Di-Et :  $\text{C}_7\text{H}_{18}\text{N}_2$ . MW, 130. B.p.  $75^\circ/20$  mm.

1 : 3-N-Tetra-Et :  $\text{C}_{11}\text{H}_{26}\text{N}_2$ . MW, 186. B.p.  $205-9^\circ$ .  $B_2\text{HCl}, \text{HgCl}_2$ : prisms from  $\text{H}_2\text{O}$ . M.p.  $124-5^\circ$ .

1 : 3-N-Diacetyl: prisms from  $\text{EtOH}$ . M.p.  $101^\circ$ . Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ , ligroin. Weak base.

1 : 3-N-Dibenzoyl: cryst. powder from  $\text{C}_6\text{H}_6$ . M.p.  $147-8^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Mod. sol. cold  $\text{C}_6\text{H}_6$ .

1 : 3-N-Dicarbomethoxyl: plates from  $\text{Et}_2\text{O}$ . M.p.  $74-5^\circ$ . Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{C}_6\text{H}_6$ , ligroin.

1 : 3-N-Dicarbomethoxyl: prisms from  $\text{Et}_2\text{O}$ . M.p.  $42^\circ$ . B.p.  $210^\circ/30$  mm. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ . Spar. sol. ligroin. Insol.  $\text{H}_2\text{O}$ .

Dipicrate: yellow leaflets.

Clarke, *J. Chem. Soc.*, 1913, **103**, 1699.Fischer, Koch, *Ber.*, 1884, **17**, 1799.Putochin, *Ber.*, 1926, **59**, 625.Ing, Manske, *J. Chem. Soc.*, 1926, 2351.**Trimethylene Glycol (1 : 3-Dihydroxypropane, propandiol-1 : 3)** $\text{C}_3\text{H}_8\text{O}_2$ 

MW, 76

Pale yellow liq. B.p.  $210-11^\circ$ ,  $109-10^\circ/12$  mm.  $D_4^{20}$  1.0597.  $n_D^{20}$  1.43983. Misc. with  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .  $\text{H}_2\text{O}_2 \rightarrow$  propionaldehyde.

$B_2\text{HNO}_3$ : viscous liq.  $D_4^{15}$  1.408. Sol.  $\text{MeOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Sol. 5 parts  $\text{EtOH}$ , 410 parts  $\text{H}_2\text{O}$ . Spar. sol.  $\text{CS}_2$ .

Me ether :  $\text{C}_4\text{H}_{10}\text{O}_2$ . MW, 90. B.p.  $153.15-153.2^\circ/768$  mm.  $D_4^{20}$  0.9434.  $n_D^{20}$  1.41259.

Phenyl ether : b.p.  $230-1^\circ$ . Formyl: b.p.  $146-7^\circ/767$  mm.  $D_4^{15}$  1.0057. Acetyl: b.p.  $162-163.5^\circ/762$  mm.  $D_4^{15}$  0.9803.

Et ether :  $\text{C}_5\text{H}_{12}\text{O}_2$ . MW, 104. B.p.  $162.1-162.2^\circ$ .  $D_4^{20}$  0.91691.  $n_D^{20}$  1.41666. Misc. with  $\text{H}_2\text{O}$ . Phenyl ether: b.p.  $328-30^\circ$ . Formyl: b.p.  $157.5-159^\circ/742$  mm.  $D_4^{15}$  0.9731. Acetyl: b.p.  $174.5-175.5^\circ$ .  $D_4^{15}$  0.9567.

Di-Et ether :  $\text{C}_7\text{H}_{16}\text{O}_2$ . MW, 132. B.p.  $140-1^\circ$ .  $D_4^{25}$  0.835. Insol.  $\text{H}_2\text{O}$ .

*Propyl ether*:  $C_6H_{14}O_2$ . MW, 118. B.p. 170–2°.  $D_4^{15}$  0.9076. *Formyl*: b.p. 174.5–176°.

*Methylene ether*: see 1 : 3-Dioxan.

*Phenyl ether*:  $C_9H_{12}O_2$ . MW, 152. B.p. 249–50°/764 mm.

*Diphenyl ether*: 1 : 3-diphenoxyp propane.  $C_{15}H_{16}O_2$ . MW, 228. Leaflets from EtOH. M.p. 61°. B.p. 338–40°. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.

*Di-o-tolyl ether*: b.p. 341–3°, 225°/28 mm.

*Di-m-tolyl ether*: plates from EtOH. M.p. 91°.

*Di-p-tolyl ether*: needles from EtOH. M.p. 94°.

*Di-1-naphthyl ether*: needles from EtOH. M.p. 103–4°.

*Di-2-naphthyl ether*: plates from AcOH. M.p. 148–9°.

*Formyl*: b.p. 195–7°/757 mm.  $D_4^{15}$  1.1405.

*Acetyl*: b.p. 202.5–204°.

*Diacetyl*: b.p. 209–10°.  $D^{19}$  1.070. Sol. 8–10 parts H<sub>2</sub>O.

*Dibenzoyl*: prisms or needles from pet. ether. M.p. 57.5° (53°).

*Di-p-toluenesulphonyl*: m.p. 93–4°.

*Diphenylurethane*: m.p. 137–137.5°.

*Di-1-naphthylurethane*: m.p. 164°.

Gattermann, *Ann.*, 1907, **357**, 379.

Henry, *Chem. Zentr.*, 1899, I, 968; 1907, I, 1314.

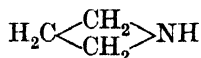
Blechta, *Chem. Abstracts*, 1922, **16**, 2991.

Rayner, *J. Soc. Chem. Ind.*, 1926, **45**, 265T, 287T.

Werkmann, Gillen, *Journal of Bacteriology*, 1932, **23**, 167.

Palomaa, *Chem. Zentr.*, 1913, II, 1959.

### Trimethyleneimine (Azetidine)



$C_3H_7N$  MW, 57

Liq. with ammoniacal odour. B.p. 63°/748 mm.  $D_{20}^{20}$  0.8436. Misc. with H<sub>2</sub>O, EtOH.

$B.HAuCl_4$ : golden-yellow cryst. M.p. 192°. Spar. sol. cold H<sub>2</sub>O.

$B_2H_2PtCl_6$ : orange-yellow needles. M.p. 203° decomp.

*Picrate*: yellow needles. M.p. 166–7°.

*N-Phenyl*: trimethylene-aniline. Oil. B.p. 242–5°, 130–2°/16 mm.

Howard, Marckwald, *Ber.*, 1899, **32**, 2032.

Scholtz, *ibid.*, 2255.

### Trimethylene iodide.

See 1 : 3-Di-iodopropane.

### Trimethylene iodohydrin.

See 3-Iodopropyl Alcohol.

### Trimethylene methylene dioxide.

See 1 : 3-Dioxan.

### Trimethylene oxide



$C_3H_6O$  MW, 58

Liq. with pleasant aromatic odour. B.p. 47.8°.  $D_4^{25}$  0.8930.  $n_D^{25}$  1.3897. Misc. with H<sub>2</sub>O.

Allen, Hibbert, *J. Am. Chem. Soc.*, 1934, **56**, 1399.

Derick, Bissell, *J. Am. Chem. Soc.*, 1916, **38**, 2478.

### 2 : 6-Trimethylenepiperidine.

See Granatanine.

### 2 : 5-Trimethylenepyrrolidine.

See Nortropene.

### Trimethylene sulphide



$C_3H_6S$  MW, 74

Mobile liq. with disagreeable odour. B.p. 93.8–94.2°/752 mm.  $D_4^{25}$  1.0284.  $n_D^{25}$  1.5059.

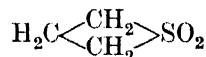
*Dimethiodide*: needles. M.p. 98.5–99.5°.

$B.HgCl_2$ : cryst. Decomp. at 93–5°. Spar. sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

Bost, Conn, *Oil and Gas Journal*, 1933, **32**, No. 3, 17.

Grishkewitsch-Trochimowski, *Chem. Zentr.*, 1923, III, 773.

### Trimethylene sulphone



$C_3H_6O_2S$  MW, 106

Needles from H<sub>2</sub>O, MeOH–Et<sub>2</sub>O, or Et<sub>2</sub>O–pet. ether. M.p. 75.5–76°. Sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O.

See second reference above.

### Trimethylene-trinitroamine.

See Hexogen.

### Trimethylethylene.

See 2-Methylbutylene-2.

### Trimethylethylene chlorohydrin.

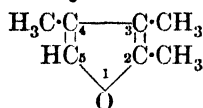
See 3-Chloro-*tert*.-amyl Alcohol.

### 1 : 1 : 2-Trimethyl-2-ethylhydracrylic Acid.

See 2-Hydroxy-1 : 1 : 2-trimethyl-*n*-valeric Acid.

### 2 : 3 : 5-Trimethyl-4-ethylpyrrole.

See Phyllopyrrole.

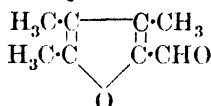
**2 : 3 : 4-Trimethylfuran** $\text{C}_7\text{H}_{10}\text{O}$ 

MW, 110

Liq. B.p. 54–5°/57 mm.

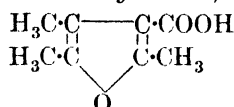
Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, 15, 1116.**2 : 3 : 5-Trimethylfuran** (2 : 4 : 5-Trimethylfuran).

B.p. 114°/720 mm., 51.5°/62 mm.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, 15, 1115.**Trimethylfuran-carboxylic Acid.**See Trimethyl- $\beta$ -furoic Acid and Trimethyl-pyromucic Acid.**3 : 4 : 5-Trimethylfurfural** $\text{C}_8\text{H}_{10}\text{O}_2$ 

MW, 138

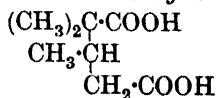
Cryst. M.p. 31–2°. B.p. 68°/0.3 mm.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, 15, 1117.**2 : 4 : 5-Trimethyl- $\beta$ -furoic Acid** (2 : 4 : 5-Trimethylfuran-3-carboxylic acid) $\text{C}_8\text{H}_{10}\text{O}_3$ 

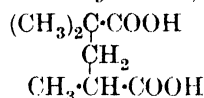
MW, 154

Cryst. from pet. ether. M.p. 131–2°. Sol. most org. solvents except pet. ether. Spar. sol.  $\text{H}_2\text{O}$ .*Et ester*:  $\text{C}_{10}\text{H}_{14}\text{O}_3$ . MW, 182. B.p. 100–5°/12 mm.Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, 15, 1114.**Trimethylglucose.**

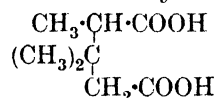
See under Glucose.

**1 : 1 : 2-Trimethylglutaric Acid** (1 : 1 : 2-Trimethylpropane-1 : 3-dicarboxylic acid, 2 : 3-dimethylbutane-1 : 3-dicarboxylic acid) $\text{C}_8\text{H}_{14}\text{O}_4$ 

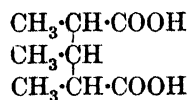
MW, 174

Cryst. M.p. 112°. Sol.  $\text{H}_2\text{O}$ , most org. solvents.*Anhydride*: prisms from pet. ether. M.p. 39°.*Monoanilide*: plates from EtOH.Aq. M.p. 155°. Sol. most org. solvents.Perkin, Thorpe, *J. Chem. Soc.*, 1897, 71, 1187.**1 : 1 : 3-Trimethylglutaric Acid** (2-Methylpentane-2 : 4-dicarboxylic acid) $\text{C}_8\text{H}_{14}\text{O}_4$ 

MW, 174

Leaflets from  $\text{H}_2\text{O}$ . M.p. 97° (95°). Sol. usual org. solvents. Less sol.  $\text{H}_2\text{O}$ .  $k = 3.48 \times 10^{-5}$  at 25°. Sublimes.*Di-Et ester*:  $\text{C}_{12}\text{H}_{22}\text{O}_4$ . MW, 230. B.p. 230–1°.  $D_4^{20}$  1.012.*Anhydride*: needles from ligroin. M.p. 95–6°. B.p. 262°.*Imide*: cryst. M.p. 139°. Sublimes.*Monoanilide*: needles. M.p. 165°. Sol. EtOH. Spar. sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{C}_6\text{H}_6$ , ligroin.Auwers, Meyer, *Ber.*, 1889, 22, 2013.Auwers, *Ann.*, 1896, 292, 224; *Ber.*, 1898, 31, 2113.**1 : 2 : 2-Trimethylglutaric Acid** (2 : 2-Dimethylbutane-1 : 3-dicarboxylic acid) $\text{C}_8\text{H}_{14}\text{O}_4$ 

MW, 174

Cryst. from  $\text{C}_6\text{H}_6$ . M.p. 88–9°. Very sol. warm  $\text{H}_2\text{O}$ . Spar. sol.  $\text{C}_6\text{H}_6$ .  $k = 1.43 \times 10^{-4}$ .*Di-Et ester*: b.p. 247–9°.*Anhydride*: prisms from AcOEt–pet. ether. M.p. 87–8° (82°).*Imide*: needles from  $\text{H}_2\text{O}$ . M.p. 126°. Sol. hot  $\text{H}_2\text{O}$ .*Monoanilide*: needles from MeOH.Aq. M.p. 150–1°.Perkin, Thorpe, *J. Chem. Soc.*, 1899, 75, 65.Crossley, Renouf, *J. Chem. Soc.*, 1911, 99, 1108.Ray, *J. Am. Chem. Soc.*, 1929, 51, 930.**1 : 2 : 3-Trimethylglutaric Acid** (3-Methylpentane-2 : 4-dicarboxylic acid) $\text{C}_8\text{H}_{14}\text{O}_4$ 

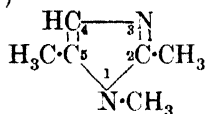
MW, 174

Cryst. from  $C_6H_6$ . M.p.  $134^\circ$ .

Ray, *J. Am. Chem. Soc.*, 1928, 50, 558; 1931, 53, 1174.

See also Michael, Ross, *J. Am. Chem. Soc.*, 1931, 53, 1175.

**1 : 2 : 5-Trimethylglyoxaline** (1 : 2 : 5-Trimethyliminazole)



$C_6H_{10}N_2$

MW, 110

Brown mobile oil.

$B.HAuCl_4$ : orange-yellow plates from dil. HCl. M.p.  $186-7^\circ$ .

*Picrate*: pale yellow needles from  $H_2O$ . M.p.  $208-9^\circ$ .

Grindley, Pyman, *J. Chem. Soc.*, 1927, 3134.

**1 : 4 : 5-Trimethylglyoxaline** (1 : 4 : 5-Trimethyliminazole).

Cryst. M.p.  $46^\circ$ . B.p.  $117^\circ/20$  mm. Sol.  $H_2O$ , EtOH,  $Et_2O$ .

$B.HCl$ : cryst. +  $H_2O$ . M.p.  $80^\circ$ , anhyd.  $199^\circ$ . Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .

$B.HNO_3$ : cryst. +  $H_2O$ . M.p.  $46^\circ$ . Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .

$B.HAuCl_4$ : yellow cryst. M.p.  $202^\circ$ .

$B_2.H_2PtCl_6$ : yellow cryst. from  $H_2O$ . M.p.  $224-5^\circ$ .

*Picrate*: yellow cryst. from  $H_2O$ . M.p.  $218^\circ$ .

*Methiodide*: needles from EtOH- $Et_2O$ . M.p.  $158^\circ$ . Sol.  $H_2O$ , EtOH. Insol.  $Et_2O$ .

Jowett, *J. Chem. Soc.*, 1905, 87, 405.

**2 : 4 : 5-Trimethylglyoxaline** (2 : 4 : 5-Trimethyliminazole).

Needles from  $Et_2O$ -ligroin. M.p.  $132.5-133^\circ$  ( $130-1^\circ$ ). Sol.  $H_2O$ , most org. solvents. Aq. sol. reacts strongly alkaline. Bitter taste.

$B.HCl$ : needles from EtOH. M.p.  $316^\circ$ .

*Picrate*: prisms from  $H_2O$ . M.p.  $163^\circ$ .

v. Pechmann, *Ber.*, 1888, 21, 1415.

Fargher, Pyman, *J. Chem. Soc.*, 1919, 115, 232.

**2 : 4 : 6-Trimethylheptanol-4.**

See Methyl-di-isobutylcarbinol.

**2 : 4 : 4-Trimethylhexanol-3.**

See Isopropyl-tert.-amylcarbinol.

**Trimethylhydracrylic Acid.**

See Hydroxydimethylbutyric Acid.

**Trimethyl - hydroxyethyl - ammonium hydroxide.**

See Choline.

**Trimethyl - hydroxyisopropyl - ammonium hydroxide.**

See  $\alpha$ -Methylcholine.

**Trimethyl - hydroxymethyl - ammonium hydroxide.**

See Formocholine.

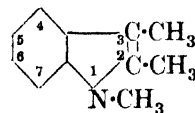
**Trimethyl - hydroxypropyl - ammonium hydroxide.**

See  $\beta$ -Methylcholine.

**Trimethyliminazole.**

See Trimethylglyoxaline.

**1 : 2 : 3-Trimethylindole**



$C_{11}H_{13}N$

MW, 159

Leaflets. M.p.  $18^\circ$ . B.p.  $283-4^\circ/750$  mm. Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Mod. sol. hot  $H_2O$ . Sol. conc. HCl.

*Picrate*: dark red needles from  $C_6H_6$ . M.p.  $150^\circ$ .

Degen, *Ann.*, 1886, 236, 160.

Ciamician, Piccinini, *Ber.*, 1896, 29, 2470.

**1 : 2 : 5-Trimethylindole.**

Leaflets from EtOH.Aq. M.p.  $56-7^\circ$ . Very sol. ligroin. Volatile in steam.

Bayer, D.R.P., 128,660, (*Chem. Zentr.*, 1902, I, 610); 137,117, (*Chem. Zentr.*, 1903, I, 109).

**2 : 3 : 5-Trimethylindole.**

Leaflets from EtOH. M.p.  $121.5^\circ$ . B.p.  $297^\circ$ . Sol. EtOH,  $CHCl_3$ , ligroin. Spar. sol.  $H_2O$ . Sol. conc. HCl. Volatile in steam.  $FeCl_3$  in AcOH  $\rightarrow$  green  $\rightarrow$  blue col.

*Picrate*: brownish-red needles from EtOH. M.p.  $189^\circ$  ( $177-80^\circ$ ).

N-Nitroso: golden-yellow needles from EtOH. M.p.  $73^\circ$ . Very sol. EtOH, AcOH. Spar. sol.  $H_2O$ .

Wolff, *Ber.*, 1888, 21, 3361.

Grgin, *Monatsh.*, 1906, 27, 739.

**2 : 3 : 7-Trimethylindole.**

Leaflets from EtOH. M.p.  $79^\circ$ . B.p.  $282-3^\circ$ . *Picrate*: purplish-red needles from  $C_6H_6$ -ligroin. M.p.  $152^\circ$ .

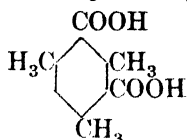
Wolff, *Ber.*, 1888, 21, 3362.

**2 : 4 : 7-Trimethylindole.**

Liq. B.p.  $158-9^\circ/13$  mm.

v. Braun, Bayer, Blessing, *Ber.*, 1924, 57, 402.

**2 : 4 : 6-Trimethylisophthalic Acid**  
(2 : 4 : 6-Trimethylbenzene-1 : 3-dicarboxylic acid, mesitylene-2 : 6-dicarboxylic acid)



$C_{11}H_{12}O_4$  MW, 208

Cryst. from AcOEt. M.p. 289° (283°). Sol. usual org. solvents.

*Dinitrile*:  $C_{11}H_{10}N_2$ . MW, 170. Prisms from EtOH.Aq. M.p. 142°.

Johnson, Fuson, *J. Am. Chem. Soc.*, 1934, 56, 1418.

Küster, Stallberg, *Ann.*, 1894, 278, 219.

**1 : 3 : 9-Trimethylisoxanthine.**

See Isocaffeine.

**Trimethylketol** (Dimethylacetylcarbinol, 2-methyl-2-butanolone-3, 3-keto-tert.-amyl alcohol)



$C_5H_{10}O_2$  MW, 102

Liq. with sweet odour. B.p. 141–2°, 50°/18 mm.  $D_4^{20}$  0.9632.

*Me ether*: oxime, prisms from ligroin. M.p. 92–3°. B.p. 190°/742 mm. Sol.  $H_2O$ , EtOH. Less sol. ligroin. Volatile in steam.

*Oxime*: leaflets from ligroin. M.p. 85–6° (96–100°).

*Semicarbazone*: cryst. from  $CHCl_3$ -pet. ether. M.p. 165°.

*Phenylhydrazone*: plates from EtOH. M.p. 83–5°.

Faworsky, *J. prakt. Chem.*, 1913, 88, 662.

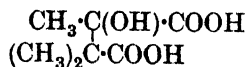
Diels, Johlin, *Ber.*, 1911, 44, 405.

Schmidt, Austin, *Ber.*, 1902, 35, 3724.

**Trimethyl-lactic Acid.**

See Hydroxydimethylbutyric Acid.

**Trimethylmalic Acid** (Hydroxytrimethylsuccinic acid)



$C_7H_{12}O_5$  MW, 176

Plates from  $H_2O$ , prisms from AcOEt. M.p. 159–60° (153–5°). M.p. depends on rate of heating. Very sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $Me_2CO$ . Sol. AcOEt. Insol.  $C_6H_6$ , ligroin.  $k = 9.4 \times 10^{-4}$  at 25°.

*Di-Et ester*:  $C_{11}H_{20}O_5$ . MW, 232. B.p. 122–3°/9 mm.  $D_{18}^{20}$  1.066.

*Anhydride*: acetyl, needles from boiling ligroin. M.p. 67–8°. Sol. usual solvents except  $H_2O$ , ligroin.

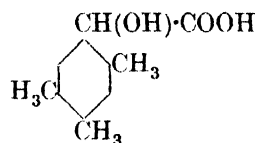
$\beta$ -Lactone: needles from  $C_6H_6$ -pet. ether. M.p. 120° (118–20°).

Auwers, v. Campenhausen, *Ber.*, 1896, 29, 1544.

Bergroth, Komppa, *Ber.*, 1896, 29, 1620.

Komppa, *Chem. Zentr.*, 1898, II, 1168; *Ber.*, 1902, 35, 534.

**2 : 4 : 5-Trimethylmandelic Acid** (2 : 4 : 5-Trimethylphenylglycollic acid)



$C_{11}H_{14}O_3$  MW, 194

Cryst. from  $C_6H_6$ . M.p. 137.5–138.5° (133–5°).

Gattermann, *Ann.*, 1906, 347, 376.

Fisher, Walling, *J. Am. Chem. Soc.*, 1935, 57, 1564.

Smith, MacMullen, *J. Am. Chem. Soc.*, 1936, 58, 633.

**2 : 4 : 6-Trimethylmandelic Acid.**

See Mesitylglycollic Acid.

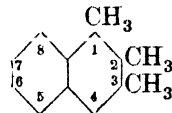
**Trimethylmethane.**

See Isobutane.

**Trimethylmethane-tricarboxylic Acid.**

See Methane-triacetic Acid.

**1 : 2 : 3-Trimethylnaphthalene**



$C_{13}H_{14}$  MW, 170

B.p. 125–30°/12 mm.

*Picrate*: light orange-yellow needles from EtOH. M.p. 142–5°.

*Styphnate*: yellow. M.p. 143–5°.

Ruzicka, Ehmann, Keller, Schütze, *Helv. Chim. Acta*, 1932, 15, 143.

**1 : 2 : 4-Trimethylnaphthalene.**

Leaflets from EtOH. M.p. 50°. B.p. 146°/12 mm.

*Picrate*: orange needles from MeOH. M.p. 147–5°.

*Styphnate*: orange-yellow needles. M.p. 123–5°.

Ruzicka, Ehmann, Tombe, Ramondt, *Helv. Chim. Acta*, 1932, 15, 145.



**1 : 2 : 5-Trimethylnaphthalene.**

M.p. 31-2°. B.p. 147-8°/11 mm.  $D_4^{25}$  1.0103.  
 $n_D^{25}$  1.6093.

*Picrate*: cryst. from EtOH. M.p. 137-8°.

*Styphnate*: cryst. from EtOH. M.p. 131°.

Ruzicka, Hosking, *Helv. Chim. Acta*,  
 1930, **13**, 1411.

Heilbron, Wilkinson, *J. Chem. Soc.*, 1930,  
 2546.

**1 : 2 : 6-Trimethylnaphthalene.**

B.p. 146°/10 mm.

*Picrate*: orange needles. M.p. 121-2°  
 (120-1°).

*Styphnate*: yellow needles. M.p. 148°.

Ruzicka, Ehmann, Cuenat, Biasotti, *Helv.  
 Chim. Acta*, 1932, **15**, 146.

**1 : 2 : 7-Trimethylnaphthalene.**

See Sapotalin.

**1 : 2 : 8-Trimethylnaphthalene.**

B.p. 152-5°/14 mm.

*Picrate*: reddish-orange needles from EtOH.  
 M.p. 139-40° (133°).

*Styphnate*: golden-yellow needles from EtOH.  
 M.p. 144-5°.

Ruzicka, Ehmann, Hartnagel, Hauss-  
 child, *Helv. Chim. Acta*, 1932, **15**, 149.

Ruzicka, Hofmann, Frei, *Helv. Chim.  
 Acta*, 1936, **19**, 391.

**1 : 3 : 5-Trimethylnaphthalene.**

Prisms from MeOH. M.p. 47° (43°). B.p.  
 139-5°/10 mm.

*Picrate*: orange-yellow needles from MeOH.  
 M.p. 141-2° (140°).

*Styphnate*: golden-yellow needles. M.p. 138°  
 (136-5°).

Heilbron, Wilkinson, *J. Chem. Soc.*, 1930,  
 2540.

Ruzicka, Ehmann, Weber, *Helv. Chim.  
 Acta*, 1932, **15**, 153.

**1 : 3 : 6-Trimethylnaphthalene.**

B.p. 140-4°/10 mm.

*Picrate*: light orange needles. M.p. 115°.

*Styphnate*: yellow needles. M.p. 148°.

Ruzicka, Ehmann, Arni, Bernasconi,  
*Helv. Chim. Acta*, 1932, **15**, 154.

**1 : 3 : 7-Trimethylnaphthalene.**

B.p. 131-3°/9 mm.  $D_4^{21}$  0.9801.  $n_D^{15}$  1.5972.

*Picrate*: orange-yellow needles from MeOH.  
 M.p. 142°.

*Styphnate*: golden-orange cryst. from MeOH.  
 M.p. 151-5°.

Ruzicka, Ehmann, Pieth, Thomann, *Helv.  
 Chim. Acta*, 1932, **15**, 155.

**1 : 3 : 8-Trimethylnaphthalene.**

Plates from MeOH. M.p. 48°.

*Picrate*: orange-red needles from EtOH.  
 M.p. 125°.

*Styphnate*: golden needles from EtOH.  
 M.p. 140-5°.

Heilbron, Wilkinson, *J. Chem. Soc.*, 1930,  
 2542.

**1 : 4 : 5-Trimethylnaphthalene.**

Leaflets from MeOH. M.p. 63°. B.p. 145°/12  
 mm.

*Picrate*: deep reddish-orange needles. M.p.  
 144-5°.

*Styphnate*: reddish-brown needles. M.p.  
 129-30°.

Ruzicka, Ehmann, Hefti, Altuna, *Helv.  
 Chim. Acta*, 1932, **15**, 156.

**1 : 4 : 6-Trimethylnaphthalene.**

B.p. 140-2°/15 mm.

*Picrate*: orange-red needles. M.p. 133°.

*Styphnate*: golden-yellow cryst. M.p. 114°.

Ruzicka, Ehmann, Addink, *Helv. Chim.  
 Acta*, 1932, **15**, 158.

**1 : 6 : 7-Trimethylnaphthalene (2 : 3 : 5-Trimethylnaphthalene).**

Cryst. from MeOH. M.p. 28°. B.p. 138°/12  
 mm.

*Picrate*: golden-orange needles from MeOH.  
 M.p. 125° (122-5°).

*Styphnate*: golden-yellow needles. M.p.  
 148-9° (146°).

Wilkinson, *J. Chem. Soc.*, 1931, 1333.

Ruzicka, Ehmann, Weisz, *Helv. Chim.  
 Acta*, 1932, **15**, 159.

Barnett, Sanders, *J. Chem. Soc.*, 1933,  
 437.

Ruzicka, *Helv. Chim. Acta*, 1936, **19**,  
 423.

**2 : 3 : 6-Trimethylnaphthalene.**

M.p. 92-3°. B.p. 263-4°, 146-8°/14 mm.

*Picrate*: orange-yellow needles from MeOH.  
 M.p. 130°.

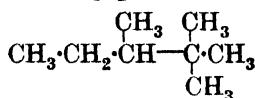
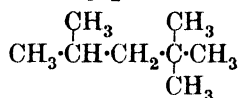
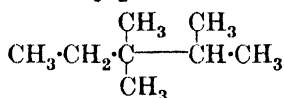
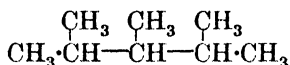
*Styphnate*: yellow needles from MeOH.  
 M.p. 165°.

Ruzicka, Ehmann, Rierink, *Helv. Chim.  
 Acta*, 1932, **15**, 160.

Collie, *J. Chem. Soc.*, 1893, **63**, 336.

**2 : 5 : 8-Trimethylnonan-5-ol.**

See Methyl-di-isoamylcarbinol.

**2 : 2 : 3-Trimethylpentane**C<sub>8</sub>H<sub>18</sub> MW, 114B.p. 110.2°. D<sub>4</sub><sup>20</sup> 0.7173. n<sub>D</sub><sup>20</sup> 1.4030.Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, 55, 2608.**2 : 2 : 4-Trimethylpentane**C<sub>8</sub>H<sub>18</sub> MW, 114F.p. -107.4°. B.p. 99.3°. D<sub>4</sub><sup>20</sup> 0.6918. n<sub>D</sub><sup>20</sup> 1.3916.Petrov, Andreev, Chapluigin, *Chem. Abstracts*, 1933, 27, 266.Edgar, Calingaert, *J. Am. Chem. Soc.*, 1929, 51, 1546.**2 : 3 : 3-Trimethylpentane**C<sub>8</sub>H<sub>18</sub> MW, 114B.p. 113.6°/760 mm. D<sub>4</sub><sup>20</sup> 0.7258. n<sub>D</sub><sup>20</sup> 1.4074.Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, 55, 2608.**2 : 3 : 4-Trimethylpentane**C<sub>8</sub>H<sub>18</sub> MW, 114B.p. 112.8°/760 mm., 111.5°/732 mm. D<sub>4</sub><sup>20</sup> 0.7197. n<sub>D</sub><sup>20</sup> 1.4045.

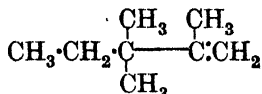
See previous reference.

**Trimethylpentanol.**

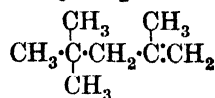
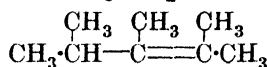
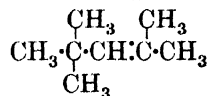
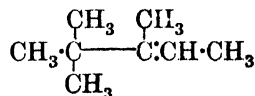
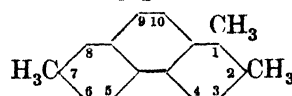
See Methyl-ethyl-tert.-butylcarbinol and Isopropyl-tert.-butylcarbinol.

**2 : 2 : 4-Trimethylpentanone-3.**

See Isopropyl-tert.-butyl Ketone.

**2 : 3 : 3-Trimethyl-1-pentene**C<sub>8</sub>H<sub>16</sub> MW, 112B.p. 108.2°/760 mm. D<sub>4</sub><sup>20</sup> 0.7363. n<sub>D</sub><sup>20</sup> 1.4178.Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, 55, 2608.

Dict. of Org. Comp.—III.

**2 : 4 : 4-Trimethyl-1-pentene**C<sub>8</sub>H<sub>16</sub> MW, 112One constituent of "di-isobutylene" (or "isodibutylene"). F.p. -93.6°. B.p. 101.2°/760 mm., 100.1°/737 mm. D<sub>4</sub><sup>20</sup> 0.7151. n<sub>D</sub><sup>20</sup> 1.4082.Tongberg, Pickens, Fenske, Whitmore, *J. Am. Chem. Soc.*, 1932, 54, 3706.Whitmore, Church, *ibid.*, 3710 (*Bibl.*).See also Petrov, Anzus, Ardrejew, *Bull. soc. chim.*, 1933, 53, 327 (*Bibl.*).**2 : 3 : 4-Trimethyl-2-pentene**C<sub>8</sub>H<sub>16</sub> MW, 112B.p. 114.3°/739 mm. n<sub>D</sub><sup>20</sup> 1.4263.Whitmore, Laughlin, *J. Am. Chem. Soc.*, 1932, 54, 4392.**2 : 4 : 4-Trimethyl-2-pentene**C<sub>8</sub>H<sub>16</sub> MW, 112One constituent of "di-isobutylene" (or "isodibutylene"). F.p. -106.5°. B.p. 104.5°/760 mm., 103.4°/737 mm. D<sub>4</sub><sup>20</sup> 0.7211. n<sub>D</sub><sup>20</sup> 1.4158.Tongberg, Pickens, Fenske, Whitmore, *J. Am. Chem. Soc.*, 1932, 54, 3706.Whitmore, Church, *ibid.*, 3710 (*Bibl.*).See also Petrov, Anzus, Ardrejew, *Bull. soc. chim.*, 1933, 53, 327.**3 : 4 : 4-Trimethyl-2-pentene**C<sub>8</sub>H<sub>16</sub> MW, 112B.p. 111.9°/760 mm. D<sub>4</sub><sup>20</sup> 0.7395. n<sub>D</sub><sup>20</sup> 1.4232.Laughlin, Whitmore, *J. Am. Chem. Soc.*, 1933, 55, 2608.**1 : 2 : 7-Trimethylphenanthrene**C<sub>17</sub>H<sub>16</sub> MW, 220  
55

Plates from EtOH. M.p. 120–1°.

*Picrate*: orange needles. M.p. 148–9°.

*Styphnate*: orange needles. M.p. 169–70°.

Haworth, Bolam, *J. Chem. Soc.*, 1932, 2250.

### 1 : 2 : 8-Trimethylphenanthrene.

Plates from EtOH. M.p. 144–5°. B.p. 210–20°/15 mm.

*Picrate*: orange-red needles from EtOH. M.p. 163°.

Haworth, Mavin, *J. Chem. Soc.*, 1932, 2723.

Ruzicka, Hosking, *Helv. Chim. Acta*, 1931, 14, 203.

### 1 : 3 : 7-Trimethylphenanthrene.

Prisms from MeOH. M.p. 68–9°.

*Picrate*: pale orange needles from EtOH. M.p. 163–4°.

*Styphnate*: yellow needles. M.p. 160–1°.

Haworth, Bolam, *J. Chem. Soc.*, 1932, 2250.

### 1 : 4 : 7-Trimethylphenanthrene.

Prisms from EtOH. M.p. 72–3°.

*Picrate*: orange needles from MeOH. M.p. 141–2°.

*Styphnate*: yellow needles. M.p. 129–30°.

Haworth, Letsky, Mavin, *J. Chem. Soc.*, 1932, 1789.

### 1 : 6 : 7-Trimethylphenanthrene.

Plates from EtOH. M.p. 123–4°.

*Picrate*: orange needles. M.p. 165–6°.

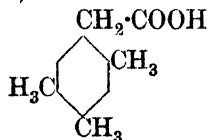
*Styphnate*: yellow needles. M.p. 111–12°.

Haworth, Bolam, *J. Chem. Soc.*, 1932, 2251.

### Trimethylphenol.

See Hemimellitenol, Hydroxy- $\psi$ -cumene, and Mesitol.

**2 : 4 : 5-Trimethylphenylacetic Acid** (5- $\psi$ -Cumylacetic acid)



$C_{11}H_{14}O_2$

MW, 178

Needles from  $H_2O$ . M.p. 128–9° (118°). Sol. usual org. solvents. Sublimes.

*Amide*:  $C_{11}H_{15}ON$ . MW, 177. Leaflets from  $H_2O$ . M.p. 174°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Sublimes.

*Nitrile*:  $C_{11}H_{13}N$ . MW, 159. M.p. 9–10°. B.p. 133–7°/4 mm.

Smith, MacMullen, *J. Am. Chem. Soc.*, 1936, 58, 632.

Willgerodt, *J. prakt. Chem.*, 1909, 80, 185.

Willgerodt, Scholz, *J. prakt. Chem.*, 1910, 81, 388.

### 2 : 4 : 6-Trimethylphenylacetic Acid.

See Mesitylacetic Acid.

### Trimethylphenylenediamine.

See under Phenylenediamine.

### Trimethylphenylglycollic Acid.

See Mesitylglycollic Acid and Trimethylmandelic Acid.

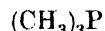
### 2 : 4 : 6-Trimethylphloroglucinol.

See 2 : 4 : 6-Trihydroxymesitylene.

### Trimethyl phosphate.

See under Phosphoric Acid.

### Trimethylphosphine



$C_3H_9P$

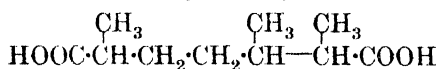
MW, 76

B.p. 40–2°. Insol.  $H_2O$ .

Cahours, Hofmann, *Ann.*, 1857, 104, 29.

Dreschel, *J. prakt. Chem.*, 1874, 10, 180.

**1 : 2 : 5-Trimethylpimelic Acid** (3-Methylheptane-2 : 6-dicarboxylic acid)



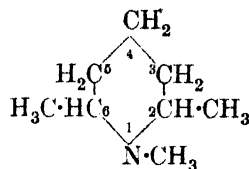
$C_{10}H_{18}O_4$

MW, 202

Oil. B.p. 213–15°/15 mm. Dist. with KOH  $\rightarrow$  2 : 3 : 6-trimethylcyclohexanone.

Zelinsky, Reformatsky, *Ber.*, 1895, 28, 2944.

### 1 : 2 : 6-Trimethylpiperidine



$C_8H_{17}N$

MW, 127

*Cis*:

Mobile liq. B.p. 50–5°/12 mm.

$B.HAuCl_4$ : m.p. 174–5°.

*Methiodide*: m.p. 275°.

Mannich, *Arch. Pharm.*, 1934, 272, 356.

### 2 : 2 : 4-Trimethylpiperidine.

B.p. 148°.  $D_{20}^{25}$  0.832. Sol. EtOH,  $Et_2O$ . Mod. sol.  $H_2O$ . Turns yellow in air.  $FeCl_3 \rightarrow$  red ppt.

$B_2HAuCl_4$ : prisms. M.p. 135°.

$B_2H_2PtCl_6$ : red cryst. M.p. 215–16° decomp.

Methiodide: prisms from  $H_2O$  or EtOH. M.p. 266° decomp.

Issoglio, *Chem. Zentr.*, 1908, II, 1444.

### 2 : 2 : 6-Trimethylpiperidine.

B.p. 138–9°.

$B_2HCl$ : m.p. 236–7°.

$B_2HAuCl_4$ : rhombohedra from  $N/HCl$ . M.p. 127–9°.

Picrate: rhombohedra from  $H_2O$ . M.p. 195–6°.

Gough, King, *J. Chem. Soc.*, 1928, 2444.

### 2 : 4 : 6-Trimethylpiperidine.

B.p. 165–6° (151–3°).  $D_4^{20}$  0.8430. Misc. with EtOH, Et<sub>2</sub>O. Spar. sol.  $H_2O$ .

$B_2HBr$ : exists in two forms. (i) Needles from  $H_2O$ . M.p. 204–9°. Sol.  $C_6H_6$ . (ii) M.p. above 270°. Spar. sol.  $C_6H_6$ .

Koenigs, Bernhart, Ibele, *Ber.*, 1907, 40, 3199, 3206.

Skita, Brunner, *Ber.*, 1916, 49, 1601.

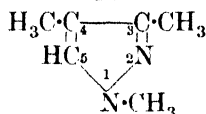
### 2 : 2 : 6-Trimethyl-γ-piperidone.

See Vinylacetoneamine.

### Trimethylpropionic Acid.

See 1 : 2-Dimethylbutyric Acid and tert.-Butylacetic Acid.

### 1 : 3 : 4-Trimethylpyrazole



$C_6H_{10}N_2$

MW, 110

Oil. B.p. 160°.  $D_4^{17.7}$  0.9567.  $n_D^{17.7}$  1.48663.

Picrate: dark yellow leaflets from  $H_2O$ . M.p. 163.5–164.5°. Very spar. sol. Et<sub>2</sub>O.

Auwers, Cauet, *J. prakt. Chem.*, 1930, 126, 202.

### 1 : 3 : 5-Trimethylpyrazole.

Needles with iodoform odour. M.p. 37°. B.p. 170°/755 mm.  $D_4^{27.8}$  0.9130.  $n_D^{27.8}$  1.45893.

$B_2HAuCl_4$ : needles +  $H_2O$  from  $H_2O$ . M.p. 91–4°.

$B_2H_2PtCl_6$ : cryst. M.p. 187–91°. Very sol.  $H_2O$ .

Picrate: m.p. 147° (144–5°).

Rojahn, Kühling, *Arch. Pharm.*, 1926, 264, 337.

Knorr, *Ann.*, 1894, 279, 232.

### 1 : 4 : 5-Trimethylpyrazole.

Oil. B.p. 176–7°.  $D_4^{17.8}$  0.9685.  $n_D^{17.8}$  1.48485.

Picrate: pale yellow needles from EtOH.

M.p. 175–6°. Mod. sol.  $H_2O$ , EtOH,  $C_6H_6$ . Spar. sol. Et<sub>2</sub>O.

Auwers, Cauet, *J. prakt. Chem.*, 1930, 126, 202.

### 3 : 4 : 5-Trimethylpyrazole.

Leaflets from  $H_2O$ . M.p. 138–9°. B.p. 232–3°/753 mm. Sol. EtOH, Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ . Volatile in steam.

$B_2HCl$ : needles. Decomp. at 265°.

$B_2H_2PtCl_6$ : yellow cryst. + 2 $H_2O$ . Decomp. about 200°.

Picrate: yellow needles from  $H_2O$  or EtOH. M.p. 239–41°.

1-N-o-Nitrobenzoyl: cryst. from EtOH. M.p. 128–9°. Sol. Et<sub>2</sub>O,  $Me_2CO$ , AcOH,  $C_6H_6$ . Spar. sol. EtOH, pet. ether.

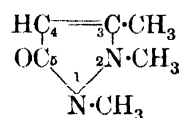
Auwers, Cauet, Wolter, *J. prakt. Chem.*, 1930, 126, 175.

v. Rothenburg, *J. prakt. Chem.*, 1895, 52, 51.

Knorr, Oettinger, *Ann.*, 1894, 279, 244.

Posner, *Ber.*, 1901, 34, 3981.

### 1 : 2 : 3-Trimethylpyrazolone-5



$C_6H_{10}ON_2$

MW, 126

B.p. 306–9°/751 mm.

$B_2H_2PtCl_6$ : prisms from EtOH.Aq. Decomp. at 197–8°.

Picrate: needles from  $H_2O$ . M.p. 211–12° decomp. Spar. sol.  $H_2O$ .

Knorr, *Ber.*, 1906, 39, 3267.

### 1 : 3 : 4-Trimethylpyrazolone-5.

Needles from  $C_6H_6$ . M.p. 133–4°. B.p. 154°/19 mm. Sol. EtOH,  $H_2O$ . Spar. sol. Et<sub>2</sub>O,  $C_6H_6$ , pet. ether.

Picrate: greenish-yellow needles from EtOH. M.p. 148°.

Auwers, Bähr, *J. prakt. Chem.*, 1927, 116, 82.

### 3 : 4 : 4-Trimethylpyrazolone-5.

Two compounds of this structure are described in the literature.

(i) Prisms from EtOH. M.p. 109.5°. Sol.  $H_2O$ , Et<sub>2</sub>O,  $Me_2CO$ ,  $CHCl_3$ , AcOH. Less sol. EtOH. Almost insol. pet. ether. No col. with  $FeCl_3$ .

Backer, *Rec. trav. chim.*, 1926, 45, 86.

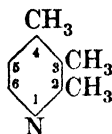
(ii) Cryst. from EtOH or H<sub>2</sub>O. M.p. 269° (262–3°).

*Acetyl*: cryst. powder. M.p. 168°.

v. Rothenburg, *J. prakt. Chem.*, 1895, 52, 43.

De, Dutt, *J. Indian Chem. Soc.*, 1930, 7, 478.

### 2 : 3 : 4-Trimethylpyridine ( $\alpha\beta\gamma$ -Collidine)



C<sub>8</sub>H<sub>11</sub>N

MW, 121

Found in low temperature coal tar. B.p. 192–3°. D<sub>20</sub> 0.9127 (0.9566). Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. KMnO<sub>4</sub> → carbocinchomeric acid. Easily decomp.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: yellow prisms from H<sub>2</sub>O. M.p. 259°.

B<sub>2</sub>HAuCl<sub>4</sub>: yellow needles from H<sub>2</sub>O. M.p. about 182–3°.

*Picrate*: yellow needles. M.p. 163–4°.

Guareschi, *Chem. Zentr.*, 1900, I, 1161.

Oparina, *Ber.*, 1931, 64, 563, 574.

### 2 : 3 : 5-Trimethylpyridine ( $\alpha\beta\beta'$ -Collidine).

Found in low temperature coal tar. B.p. 182–3°/739 mm. Spar. sol. H<sub>2</sub>O. Volatile in steam.

B<sub>2</sub>HAuCl<sub>4</sub>: needles from H<sub>2</sub>O. M.p. 146–7°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: yellow needles from dil. HCl. M.p. 227–8°.

*Picrate*: yellow needles. M.p. 183° (179°).

Komatsu, Mohri, *J. Chem. Soc. Japan*, 1931, 52, 722.

Oparina, *Ber.*, 1931, 64, 563.

### 2 : 3 : 6-Trimethylpyridine ( $\alpha\beta\alpha'$ -Collidine).

Found in low temperature coal tar. B.p. 176–8°/759 mm., 173–4°/734 mm.

B<sub>2</sub>HAuCl<sub>4</sub>: needles. M.p. 106°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: cryst. + H<sub>2</sub>O. M.p. 250–2° decomp.

*Picrate*: yellow needles. M.p. 146°.

Oparina, *Ber.*, 1931, 64, 573.

Eckert, Loria, *Monatsh.*, 1917, 38, 228, 240.

Basu, Banerjee, *J. Indian Chem. Soc.*, 1935, 12, 665.

### 2 : 4 : 5-Trimethylpyridine ( $\alpha\gamma\beta'$ -Collidine).

B.p. 165–8°. Spar. sol. H<sub>2</sub>O. KMnO<sub>4</sub> → berberonic acid.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>.H<sub>2</sub>O: red cryst. Decomp. at 205°.

B<sub>2</sub>HAuCl<sub>4</sub>: plates or needles from dil. HCl. M.p. 129–31°.

*Picrate*: orange needles. M.p. 128–31°.

Ahrens, *Ber.*, 1896, 29, 2998.

### 2 : 4 : 6-Trimethylpyridine ( $\gamma$ -Collidine, $\alpha\gamma\alpha'$ -collidine).

Found in low temperature coal tar. B.p. 171–2° (175–8°). D<sub>20</sub> 0.917.  $k = 2.05 \times 10^{-7}$  at 25°.

B<sub>2</sub>HI: prisms. Decomp. at 250°.

B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>: needles or prisms. M.p. 205°.

B<sub>2</sub>HAuCl<sub>4</sub>: needles + H<sub>2</sub>O. M.p. 53°, anhyd. 112–13°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: yellow cryst. from H<sub>2</sub>O. M.p. 223–4° decomp.

B<sub>2</sub>HCl<sub>2</sub>HgCl<sub>2</sub>: needles from H<sub>2</sub>O. M.p. 158–60°.

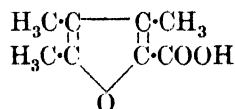
*Picrate*: yellow needles from H<sub>2</sub>O. M.p. 155–6°.

*Methoperchlorate*: prisms from H<sub>2</sub>O. M.p. 206–7°.

Hantzsch, *Ann.*, 1882, 215, 32.

Dürkopf, *Ber.*, 1888, 21, 2713.

### 3 : 4 : 5-Trimethylpyromucic Acid (3 : 4 : 5-Trimethylfuran-2-carboxylic acid, 3 : 4 : 5-trimethyl- $\alpha$ -furoic acid)



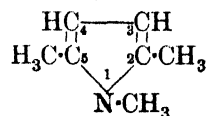
C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>

MW, 154

Cryst. from pet. ether. M.p. 185° decomp.

Reichstein, Zschokke, Syz, *Helv. Chim. Acta*, 1932, 15, 1117.

### 1 : 2 : 5-Trimethylpyrrole



C<sub>7</sub>H<sub>11</sub>N

MW, 109

B.p. 169°/746 mm. (162–4°), 55–6°/9 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Volatile in steam. Hot aq. FeCl<sub>3</sub> → deep cherry-red col.

Lukeš, *Chem. Abstracts*, 1932, 26, 4328.

Lukeš, Přeučil, *Chem. Zentr.*, 1936, I, 2082.

Knorr, *Ann.*, 1886, 236, 304.

### 2 : 3 : 4-Trimethylpyrrole.

Prisms. M.p. 39°. B.p. 71–72.5°/10 mm. Volatile in steam.

**Picrate**: yellow leaflets from EtOH. M.p. 148° (140°).

Piloty, Hirsch, *Ann.*, 1913, **395**, 66.

Fischer, Walach, *Ann.*, 1926, **450**, 114; 1926, **447**, 47.

### 2 : 3 : 5-Trimethylpyrrole.

B.p. 180°/768 mm., 79–80°/15 mm.

$B_2.Hg(HgCl_2)_4$ : m.p. 120–5° decomp. Sol. AcOH. Spar. sol.  $H_2O$ . Insol. most org. solvents.

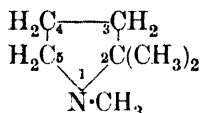
Korschun, *Ber.*, 1905, **38**, 1129.

Nenitzescu, Solomonica, *Ber.*, 1931, **64**, 1928.

Fischer, Müller, *Z. physiol. Chem.*, 1925, **148**, 155.

Piloty, Hirsch, *Ann.*, 1913, **395**, 68.

### 1 : 2 : 2-Trimethylpyrrolidine



$C_7H_{15}N$

MW, 113

Liq. B.p. 130–5°.

Lukeš, *Chem. Abstracts*, 1931, **25**, 102.

### 1 : 2 : 4-Trimethylpyrrolidine.

B.p. 111–13°.  $D^{15}_D$  0.790.

$B.HAuCl_4$ : golden-yellow ppt. M.p. 98–9°.

$B_2.H_2PtCl_6$ : orange-yellow prisms. M.p. 179–80°. Sol.  $H_2O$ .

Jacobi, Merling, *Ann.*, 1894, **278**, 9.

### 1 : 2 : 5-Trimethylpyrrolidine.

B.p. 115–16°/750 mm.  $D^{20}_D$  0.8149.  $n^{20}_D$  1.4335. Spar. sol.  $H_2O$ .

$B.HAuCl_4$ : golden-yellow cryst. M.p. about 178°.

$B_2.H_2PtCl_6$ : orange-yellow leaflets from EtOH. Decomp. at 190–210°.

**Picrate**: cryst. from EtOH. M.p. about 163°. Spar. sol.  $H_2O$ .

**Picronate**: needles. M.p. about 193°. Sol.  $H_2O$ .

**Methiodide**: cryst. from EtOH. M.p. 310°.

Knorr, Rabe, *Ber.*, 1901, **34**, 3500.

Tafel, Neugebauer, *Ber.*, 1890, **23**, 1548.

Merling, *Ann.*, 1891, **264**, 334.

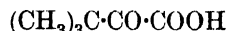
### 2 : 3 : 5-Trimethylpyrrolidine.

B.p. 126–8°.  $D^{15}_D$  0.816. Misc. with  $H_2O$ .

$B_2.H_2PtCl_6$ : orange-red prisms. M.p. 205–6° decomp.

Jacobi, Merling, *Ann.*, 1894, **278**, 13.

**Trimethylpyruvic Acid** (tert.-Butylglyoxylic acid, 1-keto-2 : 2-dimethylpropionic acid, 1-keto-tert.-butylacetic acid)



$C_6H_{10}O_3$

MW, 130

Needles from Et<sub>2</sub>O. M.p. 90–1° (82°). B.p. 189°/747 mm., 80°/15 mm. Sol. Et<sub>2</sub>O. Less sol.  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ . Ox. → pivalic acid. Volatile in steam.

**Me ester**:  $C_7H_{12}O_3$ . MW, 144. B.p. 160–2°, 69–70°/20 mm.  $D^{20}_D$  0.994. **Oxime**: cryst. from Et<sub>2</sub>O-pet. ether. M.p. 66°. **Semicarbazone**: cryst. from EtOH.Aq. M.p. 125°.

**Et ester**:  $C_8H_{14}O_3$ . MW, 158. B.p. 76–7°/20 mm., 68°/15 mm.  $D^{20}_D$  0.9716. **Oxime**: cryst. M.p. 22–3°. B.p. 131–3°/20 mm. **Semicarbazone**: cryst. from EtOH.Aq. M.p. 115°.

**Oxime**: leaflets +  $H_2O$  from EtOH. M.p. 85–6°, anhyd. 121°.

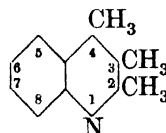
**Semicarbazone**: cryst. from  $H_2O$ . M.p. 195° (181°).

**Phenyldrazone**: yellowish needles from EtOH.Aq. M.p. 157–8° decomp.

Richard, *Ann. chim. phys.*, 1910, **21**, 360.

Glückermann, *Monatsh.*, 1889, **10**, 771.

### 2 : 3 : 4-Trimethylquinoline (3 : 4-Dimethylquinaldine)



$C_{12}H_{13}N$

MW, 171

Cryst. M.p. 92° (65°). B.p. 285°, 156–8°/12 mm.

$B.HCl$ : m.p. 274°. Spar. sol. EtOH.

$B_2.H_2PtCl_6$ : reddish-yellow. M.p. 215°.

**Methiodide**: yellow. M.p. 260°. Spar. sol. EtOH.

**Picrate**: cryst. from EtOH. M.p. 216°.

v. Braun, Gmelin, Petzold, *Ber.*, 1924, **57**, 387.

Combes, *Compt. rend.*, 1888, **106**, 143.

### 2 : 3 : 6-Trimethylquinoline (3 : 6-Dimethylquinaldine).

Cryst. from ligroin. M.p. 86–7°. B.p. 285°. Sol. Et<sub>2</sub>O. Mod. sol. EtOH. Spar. sol.  $C_6H_6$ , ligroin. Insol.  $H_2O$ .

**Picrate**: yellow leaflets from EtOH. M.p. 212° decomp.

v. Miller, *Ber.*, 1890, **23**, 2268.

**2 : 3 : 8-Trimethylquinoline** (3 : 8-Dimethylquinaldine).

Found in Californian petroleum. Plates from 50% EtOH. M.p. 55-6°. B.p. 280°/747 mm., part. decomp. Sol. most org. solvents. Spar. sol. H<sub>2</sub>O. Volatile in steam.

*B.HCl*: needles from H<sub>2</sub>O or EtOH. Darkens at 230°, decomp. at 260°.

*B.HNO<sub>3</sub>*: needles from 95% EtOH. M.p. 160-5° decomp.

*B.H<sub>2</sub>SO<sub>4</sub>*: prisms from 95% EtOH. Decomp. at about 275°.

*Picrate*: yellow cryst. from AcOH. Decomp. at 242-5°.

Poth *et al.*, *J. Am. Chem. Soc.*, 1930, **52**, 1245.

**2 : 4 : 6-Trimethylquinoline** (4 : 6-Dimethylquinaldine).

Needles + H<sub>2</sub>O from H<sub>2</sub>O + trace EtOH. M.p. 39.4°, anhyd. 65.5°. B.p. 281-2°, 146-8°/13.5 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, pet. ether. Spar. sol. H<sub>2</sub>O. Bitter taste. Sweet odour. Spar. volatile in steam.

*B.HCl*: needles from H<sub>2</sub>O. M.p. 268-72°.

*B.HBr*: needles from EtOH.Aq. M.p. about 265-70°.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: needles from EtOH. M.p. 221-2°.

*B.HAuCl<sub>4</sub>*: yellow needles. M.p. 140° decomp.

*Tartrate*: cryst. from EtOH. M.p. 172°.

*Picrate*: greenish-yellow needles from Me<sub>2</sub>CO. M.p. 200-1°. Spar. sol. hot EtOH.

*Picolonate*: yellow needles from C<sub>6</sub>H<sub>6</sub>-EtOH. M.p. about 245°.

*Methiodide*: pale yellow needles from EtOH. M.p. 245-7° (225-6°).

*Ethochloride*: needles from EtOH-Et<sub>2</sub>O. Decomp. at 247°. Very sol. H<sub>2</sub>O, EtOH.

*Ethobromide*: leaflets from EtOH. Decomp. at 246°. Very sol. H<sub>2</sub>O.

Pfitzinger, *J. prakt. Chem.*, 1888, **38**, 41.

Knoll, D.R.Ps., 363,582-3, (*Chem. Abstracts*, 1924, **18**, 991).

Bähr, *Ber.*, 1922, **55**, 1925.

Fischer, Scheibe, Müller, Merkel, *J. prakt. Chem.*, 1920, **100**, 97.

**2 : 4 : 7-Trimethylquinoline** (4 : 7-Dimethylquinaldine).

Liq. B.p. 280-1°. D<sub>20</sub> 1.0337. n<sub>D</sub><sup>20</sup> 1.59732. Forms hydrate with 1H<sub>2</sub>O, m.p. 48°.

*B.HCl*: cryst. + 2H<sub>2</sub>O. Sublimes at 310°.

*B.HBr*: cryst. + H<sub>2</sub>O. Decomp. at 351°.

*B.HI*: decomp. at 320°.

*B.HAuCl<sub>4</sub>*: m.p. 152°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: decomp. at 272°.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: cryst. + H<sub>2</sub>O. M.p. 233°.

*Picrate*: decomp. at 232°.

*Methiodide*: sublimes at 322°.

Yamaguchi, *J. Pharm. Soc. Japan*, 1924, **503**, 23.

**2 : 4 : 8-Trimethylquinoline** (4 : 8-Dimethylquinaldine).

Found in petroleum. Cryst. M.p. 42°. B.p. 269-70° (280°/746 mm.). n<sub>D</sub><sup>20</sup> 1.5855.

*B.HCl*: cryst. + 2H<sub>2</sub>O. M.p. 238°.

*B.HBr*: cryst. + 2H<sub>2</sub>O. M.p. anhyd. 251°.

*B.HI*: cryst. + H<sub>2</sub>O. M.p. 224°.

*B.HAuCl<sub>4</sub>*: m.p. 191°.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: cryst. + H<sub>2</sub>O. M.p. 263°.

*Picrate*: m.p. 193°.

*Methiodide*: decomp. at 229°.

Perrin, Bailey, *J. Am. Chem. Soc.*, 1933, **55**, 4136.

See also previous reference.

**2 : 6 : 8-Trimethylquinoline** (6 : 8-Dimethylquinaldine).

Leaflets from EtOH.Aq., prisms from pet. ether. M.p. 46°. B.p. 266-7°/780 mm., 260°/719 mm. Very sol. EtOH, Et<sub>2</sub>O, pet. ether. Insol. H<sub>2</sub>O. Volatile in steam.

*B.HCl*: prisms. M.p. 207°.

*B.HBr*: prisms. M.p. 172-3°.

*B.HI*: yellow prisms. M.p. 223-4°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: brown cryst. M.p. 206-7°.

*Picrate*: yellow prisms. M.p. 187-9°.

*d-Camphorsulphonate*: m.p. 231-2°.

Panajotow, *Ber.*, 1887, **20**, 32.

v. Miller, Plöchl, *Ber.*, 1896, **29**, 1472.

Jones, Evans, *J. Chem. Soc.*, 1911, **99**, 335.

**5 : 6 : 8-Trimethylquinoline.**

Prisms. M.p. 42-3°. B.p. 285-7°. Sol. usual solvents.

*B.HNO<sub>3</sub>*: needles. Spar. sol. H<sub>2</sub>O.

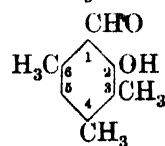
*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: prisms from EtOH.

Berend, *Ber.*, 1885, **18**, 376.

Wikander, *Ber.*, 1900, **33**, 646.

**2 : 4 : 6-Trimethylresorcinol.**

See Mesorcinol.

**3 : 4 : 6-Trimethylsalicylaldehyde** (6-Hydroxy-2 : 4 : 5-trimethylbenzaldehyde)

C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>

MW, 164

Plates from EtOH. M.p. 78-9°.

Gattermann, *Ann.*, 1906, **347**, 379.

**3 : 5 : 6-Trimethylsalicylaldehyde** (6-Hydroxy-2 : 3 : 5-trimethylbenzaldehyde).

Yellow needles from EtOH. M.p. 105–6°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH. Spar. sol. cold EtOH. Insol. H<sub>2</sub>O. Sublimes. FeCl<sub>3</sub> → blue col.

Auwers, *Ber.*, 1884, 17, 2976.

**4 : 5 : 6-Trimethylsalicylaldehyde** (6-Hydroxy-2 : 3 : 4-trimethylbenzaldehyde).

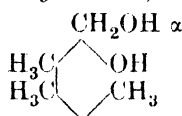
Pale yellow needles or prisms from MeOH. M.p. 77–8°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Alc. FeCl<sub>3</sub> → intense green col.

Semicarbazone: cryst. from AcOH. Does not melt below 280°.

Auwers, Ziegler, *Ann.*, 1921, 425, 276.

**Trimethylsalicylic Acid.**

See 6-Hydroxydurylic Acid and 6-Hydroxy-γ-isodurylic Acid.

**3 : 5 : 6-Trimethylsaligenin** (6-Hydroxy-2 : 3 : 5-trimethylbenzyl alcohol)

C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>

MW, 166

Needles from pet. ether. M.p. 91–2°. FeCl<sub>3</sub> in EtOH.Aq. → blue col. FeCl<sub>3</sub> in EtOH → bluish-green col.

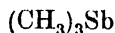
α-Me ether: C<sub>11</sub>H<sub>16</sub>O<sub>2</sub>. MW, 180. Needles from MeOH.Aq. M.p. 44–5°. Sol. usual org. solvents.

α-Acetyl: needles from AcOH.Aq. M.p. 57–8°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>, AcOH, pet. ether.

Diacetyl: needles from MeOH.Aq. M.p. 50–51.5°. Sol. usual org. solvents.

Manasse, *Ber.*, 1902, 35, 3844.

Zinke, v. Hohorst, *Ann.*, 1907, 353, 362.

**Trimethylstibine** (Antimony trimethyl)

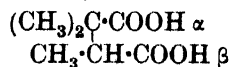
C<sub>3</sub>H<sub>9</sub>Sb

MW, 165

B.p. 82° (80.6°). Sol. EtOH, Et<sub>2</sub>O, CS<sub>2</sub>. Insol. H<sub>2</sub>O. D<sub>4</sub><sup>15</sup> 1.523. n<sub>D</sub><sup>15</sup> 1.42. Oxidises in air and takes fire. Combines with O, S, and halogens to give oxide, sulphide, and halide respectively. Pptes Ag and Hg from their sols.

Hibbert, *Ber.*, 1906, 39, 160.

Paneth, Loleit, *J. Chem. Soc.*, 1935, 371.

**Trimethylsuccinic Acid** (2-Methylbutane-2 : 3-dicarboxylic acid)

C<sub>7</sub>H<sub>12</sub>O<sub>4</sub>

MW, 160

d-.

Cryst. from H<sub>2</sub>O. M.p. 140°. [α]<sub>D</sub> + 4.83° in H<sub>2</sub>O.

Quinine salt: m.p. 197–8°. Spar. sol. H<sub>2</sub>O.

dl-.

Prisms from H<sub>2</sub>O. M.p. 152° (148–9°). Sol. 10 parts H<sub>2</sub>O at 15°. Sol. EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, CHCl<sub>3</sub>, AcOEt. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. ligroin, CS<sub>2</sub>. k = 3.22 × 10<sup>-4</sup> at 25°. Heat of comb. C<sub>v</sub> 5183 Cal. Spar. volatile in steam.

β-Et ester: C<sub>9</sub>H<sub>16</sub>O<sub>4</sub>. MW, 188. Liq. B.p. 158°/14 mm.

Di-Et ester: C<sub>11</sub>H<sub>20</sub>O<sub>4</sub>. MW, 216. B.p. 226°, 111°/14 mm. D<sub>4</sub><sup>20</sup> 0.993. n<sub>D</sub><sup>20</sup> 1.427.

β-Nitrile: C<sub>7</sub>H<sub>11</sub>O<sub>2</sub>N. MW, 141. Needles. M.p. 126°. Very sol. EtOH, CHCl<sub>3</sub>. Spar. sol. cold H<sub>2</sub>O. Insol. ligroin.

Anhydride: cryst. M.p. 38.5° (33°). B.p. 227°/746 mm., 106–7°/15 mm. Sol. usual solvents except cold H<sub>2</sub>O, ligroin. Heat of comb. C<sub>v</sub> 5884.2 Cal.

Monoanilide: needles from EtOH.Aq. M.p. 134–5°. Sol. EtOH, CHCl<sub>3</sub>. Insol. ligroin.

Mono-p-toluidide: plates from EtOH. M.p. 126°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, AcOH. Mod. sol. hot C<sub>6</sub>H<sub>6</sub>. Spar. sol. hot H<sub>2</sub>O. Insol. ligroin.

Mono-2-naphthalide: prisms from EtOH.Aq. M.p. 153°.

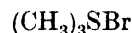
Bone, Perkin, *J. Chem. Soc.*, 1895, 67, 427.

Auwers, Oswald, *Ann.*, 1895, 285, 260, 283, 298.

Paolini, *Gazz. chim. ital.*, 1900, 30, ii, 508.

Bone, Sprankling, *J. Chem. Soc.*, 1899, 75, 858.

v. Braun, Keller, Weissbach, *Ann.*, 1931, 490, 186.

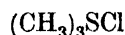
**Trimethylsulphonium bromide**

C<sub>3</sub>H<sub>9</sub>BrS

MW, 157

Cryst. from H<sub>2</sub>O. Decomp. at 172°. Reacts neutral in aq. sol.

Steinkopf, Müller, *Ber.*, 1923, 56, 1929.

**Trimethylsulphonium chloride**

C<sub>3</sub>H<sub>9</sub>ClS

MW, 112.5

Cryst. Decomp. at 100°. Very sol. EtOH. Very hygroscopic.

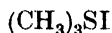
Blättler, *Monatsh.*, 1919, 40, 420.



**Trimethylsulphonium iodide**

872

**Trimethylsulphonium iodide**



$\text{C}_3\text{H}_9\text{IS}$

MW, 204

Cryst. from EtOH. Decomp. at 203–7°.

Steinkopf, Müller, *Ber.*, 1923, 56, 1928.

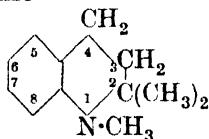
**Trimethyltetrahydrobenzaldehyde.**

See Cyclocitral.

**1 : 1 : 6 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydronaphthalene.**

See Ionene.

**1 : 2 : 2 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline**



$\text{C}_{12}\text{H}_{17}\text{N}$

MW, 175

B.p. 269–70°/745 mm.  $n_D^{20}$  1.5823. Volatile in steam.

Picrate : m.p. 178°.

Freund, Richard, *Ber.*, 1909, 42, 1112.

**1 : 2 : 4 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

B.p. about 250°/759 mm.

Picrate : yellow prisms. M.p. 126–7°.

Methiodide : cryst. from EtOH. M.p. 215°.

Ciamician, Piccinini, *Ber.*, 1896, 29, 2468.

Fischer, Meyer, *Ber.*, 1890, 23, 2633.

**1 : 2 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

Liq. B.p. 242–5°.

Doebner, v. Miller, *Ber.*, 1883, 16, 2470.

**1 : 6 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

$B, HI$  : prisms from MeOH–Et<sub>2</sub>O. M.p. 164–5°.

Ewins, *J. Chem. Soc.*, 1913, 103, 104.

**2 : 2 : 4 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

Needles. M.p. 41°. B.p. 119–22°/10 mm.  $D_4^{25}$  0.9531.  $n_D^{25}$  1.53592.

Reddelien, Thurm, *Ber.*, 1932, 65, 1520.

**2 : 4 : 6 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

B.p. 265–6°/758 mm. (261.5°/762.5 mm.).  $D_4^{21}$  0.97548.  $n_D^{21}$  1.544354.

Yamaguchi, *Chem. Abstracts*, 1927, 21, 2696.

**2 : 4 : 6-Trimethyl-5 : 6 : 7 : 8-tetra - hydroquinoline**

**2 : 4 : 7 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

B.p. 269–70°.  $D_4^{20}$  0.9857.  $n_D^{20}$  1.55591.

See previous reference.

**2 : 4 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

B.p. 260–1°.  $D_4^{20}$  0.98639.  $n_D^{20}$  1.55598.

Yamaguchi, *Chem. Abstracts*, 1927, 21, 2696.

**2 : 6 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

Plates. M.p. 50–51°. B.p. 260–1°/780 mm., 142–3°/14 mm.

$B, HCl$  : prisms from H<sub>2</sub>O. M.p. 208–9°.

$B, HBr$  : prisms. M.p. 222–3°.

$B_2, H_2PtCl_6$  : brown prisms. M.p. 210°.

Picrate : yellow prisms from EtOH. M.p. 179°.

$N$ -Acetyl : needles from EtOH. M.p. 108–9°.

$N$ -Benzoyl : prisms from EtOH. M.p. 143°.

$N$ -Nitroso : yellowish needles from EtOH. M.p. 68–9°.

Jones, Evans, *J. Chem. Soc.*, 1911, 99, 335.

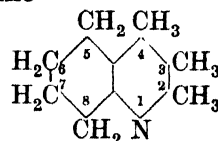
**5 : 6 : 8 - Trimethyl - 1 : 2 : 3 : 4 - tetra - hydroquinoline.**

Oil. B.p. 287–90°.

$B, HCl$  : needles. M.p. about 238° decomp. Mod. sol. H<sub>2</sub>O.

Wikander, *Ber.*, 1900, 33, 648.

**2 : 3 : 4 - Trimethyl - 5 : 6 : 7 : 8 - tetra - hydroquinoline**



$\text{C}_{12}\text{H}_{17}\text{N}$

MW, 175

B.p. 145–7°/13 mm. In moist air → dihydrate, m.p. 32°.

$B, HCl$  : m.p. 174°. Hygroscopic.

Methiodide : m.p. 125–6°.

Picrate : m.p. 147°. Spar. sol. EtOH.

v. Braun, Gmelin, Petzold, *Ber.*, 1924, 57, 387.

**2 : 4 : 6 - Trimethyl - 5 : 6 : 7 : 8 - tetra - hydroquinoline.**

B.p. 256–7°.  $D_4^{21}$  0.9845.  $n_D^{21}$  1.53007.

Yamaguchi, *Chem. Abstracts*, 1927, 21, 2696.

**2 : 4 : 7 - Trimethyl - 5 : 6 : 7 : 8 - tetra - hydroquinoline.**

Cryst. M.p. 20-1°. B.p. 259-60°.  $D_4^{20}$  0.97559.  $n_D^{20}$  1.52111.

See previous reference.

**2 : 4 : 8 - Trimethyl - 5 : 6 : 7 : 8 - tetra - hydroquinoline.**

B.p. 251-2°.  $D_4^{20}$  0.99196.  $n_D^{20}$  1.53575.

Yamaguchi, *Chem. Abstracts*, 1927, 21, 2696.

**2 : 6 : 7 - Trimethyl - 5 : 6 : 7 : 8 - tetra - hydroquinoline.**

B.p. 239-40°/752 mm.  $n_D^{21}$  1.5212.

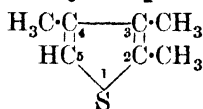
$B_2H_2PtCl_6$ : m.p. 187°.

Picrate: cryst. from EtOH.Aq. M.p. 105°.

Basu, *Ann.*, 1934, 514, 295.

**1 : 1 : 4-Trimethyltetramethylene Glycol.**

See 2-Methylhexandiol-2 : 5.

**2 : 3 : 4-Trimethylthiophene**

$C_7H_{10}S$

MW, 126

Liq. B.p. 160-3°.

v. Meyer, *Die Thiophengruppe*, 60.

Zelinsky, *Ber.*, 1887, 20, 2025.

**2 : 3 : 5-Trimethylthiophene.**

B.p. 163-5°/746 mm.  $D_4^{20}$  0.9753.  $n_D^{20}$  1.5131.

Youtz, Perkins, *J. Am. Chem. Soc.*, 1929, 51, 3514.

**Trimethylthiourea.**

See under Thiourea.

 **$\omega$ -Trimethyltoluene.**

See tert.-Butylbenzene.

**1 : 1 : 2-Trimethyltricarballic Acid.**

See Camphoronic Acid.

**2 : 4 : 6-Trimethyl-1 : 3 : 5-trioxan.**

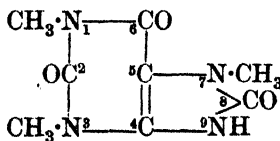
See Paraldehyde.

**1 : 3 : 5-Trimethyluracil.**

See under Thymine.

**Trimethylurea.**

See under Urea.

**1 : 3 : 7-Trimethyluric Acid**

$C_8H_{10}O_3N_4$

MW, 210

Needles from  $H_2O$ . M.p. 345° (335°) decomp.  $k = 2.9 \times 10^{-5}$  at 25°. Spar. sol. cold  $H_2O$ , EtOH, Et<sub>2</sub>O.

Biltz, Pardon, *J. prakt. Chem.*, 1934, 140, 220.

Biltz, Heyn, *Ann.*, 1917, 413, 179.

**1 : 3 : 9-Trimethyluric Acid.**

Plates. Decomp. at 347° (340°). Sol.  $NH_3$ . Spar. sol. EtOH,  $CHCl_3$ . Insol.  $H_2O$ . Sol. 30 parts boiling  $H_2O$ .  $k = 1.3 \times 10^{-6}$  at 25°. Reduces  $NH_3 \cdot AgNO_3$  on boiling.

Biltz, Strufe, *Ann.*, 1921, 423, 242.

Biltz, Pardon, *Ann.*, 1935, 515, 241; *Ber.*, 1930, 63, 2878.

**1 : 7 : 9-Trimethyluric Acid.**

Needles from EtOH or  $H_2O$ . M.p. 345° (338°). Mod. sol. hot  $H_2O$ . Spar. sol. hot EtOH,  $CHCl_3$ .  $k = 1.00 \times 10^{-4}$  at 25°. Sublimes.

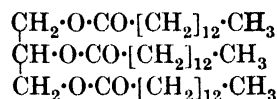
Biltz, Krzikalla, *Ann.*, 1921, 423, 180.

**3 : 7 : 9-Trimethyluric Acid.**

Needles. M.p. 373-5°.  $k = 1.7 \times 10^{-6}$  at 25°.

Biltz, Damm, *Ann.*, 1917, 413, 186.

Biltz, Pardon, *Ber.*, 1930, 63, 2876.

**Trimyristin (Glycerol trimyristate)**

$C_{45}H_{86}O_6$

MW, 722

Found in nutmegs. Exists in polymorphic forms. (i) Unstable. M.p. 32.1°. (ii) Unstable. M.p. 41.8°. (iii) Stable. Plates from Et<sub>2</sub>O. M.p. 56.5°.

Sol. Et<sub>2</sub>O,  $CHCl_3$ ,  $C_6H_6$ , pet. ether. Spar. sol. EtOH,  $CS_2$ , ligroin.  $D_4^{20}$  0.8848.  $n_D^{20}$  1.44285. Heat of comb.  $C_p$  6650 Cal.

Beal, *Organic Syntheses*, Collective Vol. I, 524.

Bömer, Engel, *Chem. Abstracts*, 1929, 23, 4676.

**2 : 4 : 6-Trinitroacetanilide.**

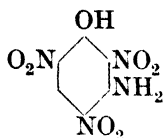
See under Picramide.

**Trinitroaminoanisole.**

See under Trinitroaminophenol.

**Trinitroaminophenetole.**

See under Trinitroaminophenol.

2 : 4 : 6-Trinitro-*m*-aminophenol $C_6H_4O_7N_4$ 

MW, 244

Cryst. from  $C_6H_6$ . M.p. 178–9°.

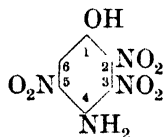
*Me ether* : 2 : 4 : 6-trinitro-*m*-aminoanisole, 2 : 4 : 6-trinitro-*m*-anisidine.  $C_7H_6O_7N_4$ . MW, 258. Cryst. from MeOH. M.p. 131°.

*Et ether* : 2 : 4 : 6-trinitro-*m*-aminophenetole, 2 : 4 : 6-trinitro-*m*-anisidine.  $C_8H_8O_7N_4$ . MW, 272. Cryst. from AcOH. M.p. 107°.

*N-Me* :  $C_7H_6O_7N_4$ . MW, 258. Yellow cryst. M.p. 158°.

v. Duin, v. Lennep, *Rec. trav. chim.*, 1920, 39, 149.

Flürscheim, D.R.P., 243,079, (*Chem. Zentr.*, 1912, I, 620).

2 : 3 : 5-Trinitro-*p*-aminophenol $C_6H_4O_7N_4$ 

MW, 244

*Me ether* : 2 : 3 : 5-trinitro-*p*-aminoanisole, 2 : 3 : 5-trinitro-*p*-anisidine. Reddish-brown needles from  $H_2O$ . M.p. 127–8°. Sol.  $Me_2CO$ ,  $PhNO_2$ . Mod. sol.  $Et_2O$ ,  $C_6H_6$ . *N-Acetyl* : needles from AcOH.Aq. or  $Me_2CO$ .Aq. M.p. 242°. Spar. sol. boiling  $H_2O$ . Insol.  $Et_2O$ ,  $C_6H_6$ . *N-Benzoyl* : m.p. 220–30°. *N-p-Toluenesulphonyl* : needles from  $Me_2CO$ . M.p. 217°.

*Et ether* : 2 : 3 : 5-trinitro-*p*-aminophenetole, 2 : 3 : 5-trinitro-*p*-phenetidine. Red needles with green metallic reflex from MeOH. M.p. 126–7°. Sol.  $Me_2CO$ , hot EtOH, AcOH,  $PhNO_2$ . *N-Acetyl* : needles. M.p. about 245°. Sol.  $Me_2CO$ , AcOH. Spar. sol. hot EtOH,  $C_6H_6$ .

*N-Acetyl* : brownish cryst. from AcOH. M.p. 191–2°. Very sol. EtOH, AcOH.

Reverdin, Meldola, *J. prakt. Chem.*, 1913, 88, 787.

Reverdin, Fürstenberg, *ibid.*, 323.

Reverdin, *Ber.*, 1911, 44, 2364.

2 : 3 : 6-Trinitro-*p*-aminophenol.

Red needles from AcOH. Decomp. about 145°.

*Me ether* : 2 : 3 : 6-trinitro-*p*-aminoanisole, 2 : 3 : 6-trinitro-*p*-anisidine. Dark red cryst. from EtOH. M.p. 138–9°. *N-Acetyl* : needles

from EtOH. M.p. 194°. *N-Benzoyl* : needles from AcOH.Aq. M.p. 205°.

*N-Acetyl* : yellow needles from AcOH. M.p. 178–9° decomp. Sol. hot EtOH. Spar. sol. hot  $H_2O$ .

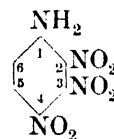
*N-Propionyl* : yellowish-brown needles from AcOH. M.p. 178–9°.

Meldola, Reverdin, *J. Chem. Soc.*, 1913, 103, 1484.

Meldola, Hay, *J. Chem. Soc.*, 1909, 95, 1380.

Meldola, Kuntzen, *J. Chem. Soc.*, 1910, 97, 455.

## 2 : 3 : 4-Trinitroaniline

 $C_6H_4O_6N_4$ 

MW, 228

*N-Di-Me* :  $C_8H_8O_6N_4$ . MW, 256. Orange rhombohedra from  $Me_2CO$  or  $C_6H_6$ . M.p. 154°.  $D^{17} 1.551$ .

v. Romburgh, *Rec. trav. chim.*, 1887, 6, 253.

## 2 : 4 : 5-Trinitroaniline.

Pale yellow needles from AcOH.

*N-Di-Me* : red cryst. from  $Me_2CO$ - $C_6H_6$ . M.p. 196°.  $D^{17} 1.585$ .

Witt, Witte, *Ber.*, 1908, 41, 3095.

See also previous reference.

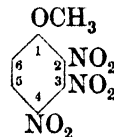
## 2 : 4 : 6-Trinitroaniline.

See Picramide.

## Trinitroanisidine.

See under Trinitroaminophenol.

## 2 : 3 : 4-Trinitroanisole

 $C_7H_5O_7N_3$ 

MW, 243

Yellow leaflets from EtOH. M.p. 155°. Sol.  $Me_2CO$ . Spar. sol. EtOH,  $C_6H_6$ . Very spar. sol. ligroin.

Vermeulen, *Chem. Zentr.*, 1912, I, 724.

Meldola, Eyre, *J. Chem. Soc.*, 1902, 81, 993.

## 2 : 3 : 5-Trinitroanisole.

Leaflets from EtOH or  $HNO_3$ . M.p. 106.8° (104°).  $D^{16} 1.618$ .

Blanksma, *Rec. trav. chim.*, 1904, 23, 111.

**2 : 4 : 5-Trinitroanisole.**

Cryst. from EtOH. M.p. 106-7°. Spar. sol. ligroin.

Vermeulen, *Rec. trav. chim.*, 1912, **31**, 102.

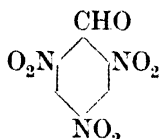
**2 : 4 : 6-Trinitroanisole.**

See under Picric Acid.

**3 : 4 : 5-Trinitroanisole.**

Cryst. M.p. 119-20°.

Vermeulen, *Rec. trav. chim.*, 1912, **31**, 103.

**2 : 4 : 6-Trinitrobenzaldehyde**

$C_7H_3O_7N_3$

MW, 241

Plates from  $C_6H_6$ . M.p. 119°. Conc.  $NH_3$ . Aq. in EtOH  $\longrightarrow$  1 : 3 : 5-trinitrobenzene.

*Oxime*: cryst. from EtOH. M.p. 158°. Sol. EtOH,  $Me_2CO$ .

*Semicarbazone*: pale yellow leaflets from AcOH. M.p. 214° decomp.

*Phenylhydrazone*: reddish-brown needles. M.p. 202°. Sol. hot  $Me_2CO$ . Spar. sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

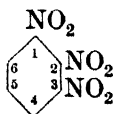
*p-Nitrophenylhydrazone*: light red needles from hot  $Me_2CO$ . M.p. 247°. Mod. sol. AcOEt,  $PhNO_2$ . Spar. sol. EtOH,  $CHCl_3$ , AcOH.

Sachs, Everding, *Ber.*, 1902, **35**, 1236; D.R.P., 121,745, (*Chem. Zentr.*, 1901, II, 69).

Secareanu, *Bull. soc. chim.*, 1932, **51**, 591; *Ber.*, 1931, **64**, 837.

**2 : 4 : 6-Trinitrobenzanilide.**

See under Picramide.

**1 : 2 : 3-Trinitrobenzene**

$C_6H_3O_6N_3$

MW, 213

Yellowish needles or prisms from MeOH. M.p. 127.5° (121°). Sol. 10 parts boiling EtOH.

Borsche, *Ber.*, 1923, **56**, 1500.

Körner, Contardi, *Atti accad. Lincei*, 1914, **23**, II, 464.

**1 : 2 : 4-Trinitrobenzene.**

Pale yellow prisms from MeOH.Aq. M.p. 61-2° (57.5°). Very sol.  $C_6H_6$ . Sol. EtOH,

MeOH,  $Et_2O$ ,  $CHCl_3$ . Heat of comb.  $C_p$  678.5 Cal.

Körner, Contardi, *Atti accad. Lincei*, 1914, **23**, I, 634.

Borsche, *Ber.*, 1923, **56**, 1498.

**1 : 3 : 5-Trinitrobenzene.**

Exists in dimorphous forms. Cryst. from EtOH or  $HNO_3$ . M.ps. 122.48° and 61°. Sol. MeOH,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ ,  $CS_2$ . Sol. 2500 parts cold  $H_2O$ . Heat of comb.  $C_p$  663.8 Cal.

Heinemann, E.P., 102,216, (*Chem. Abstracts*, 1917, **11**, 889).

Radcliffe, Pollitt, *J. Soc. Chem. Ind.*, 1921, **40**, 45, 90r.

Drummond, *J. Soc. Chem. Ind.*, 1922, **41**, 338r.

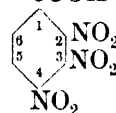
Desvergnès, *Chimie et industrie*, 1931, **25**, 3, 291 (*Bibl.*).

Secareanu, *Bull. soc. chim.*, 1932, **51**, 591.

Clarke, Hartman, *Organic Syntheses*, Collective Vol. I, 526.

**2 : 3 : 4-Trinitrobenzoic Acid**

COOH



$C_7H_3O_8N_3$

MW, 257

Prisms from  $H_2O$  or  $C_6H_6$ . M.p. 202-3°. Sol. EtOH,  $Me_2CO$ . Mod. sol.  $H_2O$ ,  $C_6H_6$ . Insol. pet. ether. Explodes when heated in quantities greater than 1 gm.

*Et ester*:  $C_9H_7O_8N_3$ . MW, 285. Needles. M.p. 79-80°. Sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ ,  $Et_2O$ . Spar. sol. pet. ether.

Giua, *Gazz. chim. ital.*, 1915, **45**, i, 348.

**2 : 3 : 5-Trinitrobenzoic Acid.**

Leaflets +  $2H_2O$ . from  $H_2O$ . M.p. 82°, anhyd. 171°.

Körner, Contardi, *Atti accad. Lincei*, 1915, **24**, I, 893.

**2 : 3 : 6-Trinitrobenzoic Acid.**

Fine white needles +  $2H_2O$  from  $H_2O$ . M.p. 55°, anhyd. 160° decomp. Above 160° or by boiling with  $H_2O \longrightarrow$  1 : 2 : 4-trinitrobenzene.

Körner, Contardi, *Atti accad. Lincei*, 1916, **25**, II, 348.

**2 : 4 : 5-Trinitrobenzoic Acid.**

Plates from  $H_2O$ . M.p. 194.5° decomp. Sol. EtOH,  $Et_2O$ . Mod. sol.  $C_6H_6$ . Spar. sol. pet. ether.

*Me ester*:  $C_8H_5O_8N_3$ . MW, 271. Cryst. from MeOH. M.p. 102°. Sol. usual org. solvents.

*Et ester*:  $C_9H_7O_8N_3$ . MW, 285. Leaflets from EtOH.Aq. M.p. 84°. Sol.  $Me_2CO$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. pet. ether.

Giua, *Gazz. chim. ital.*, 1915, 45, i, 350.

### 2 : 4 : 6-Trinitrobenzoic Acid.

Rhomboheda from  $H_2O$ . M.p. 228.7° (210°). Above m.p. or with conc.  $NH_3$ .Aq. in EtOH  $\rightarrow$  1 : 3 : 5-trinitrobenzene.

*Me ester*: orange-yellow plates from EtOH.Aq. M.p. 160-1°.

*Et ester*: orange-yellow plates. M.p. 156-7°.

*Propyl ester*:  $C_{10}H_9O_8N_3$ . MW, 299. Plates. M.p. 145-6°.

*Isopropyl ester*: yellow plates. M.p. 154-5°.

*Butyl ester*:  $C_{11}H_{11}O_8N_3$ . MW, 313. Plates. M.p. 125-6°.

*Isobutyl ester*: m.p. 127-8°.

*n-Amyl ester*:  $C_{12}H_{13}O_8N_3$ . MW, 327. M.p. 124-5°.

*Isocamyl ester*: m.p. 134-5°.

*Phenyl ester*:  $C_{13}H_7O_8N_3$ . MW, 333. Cryst. from EtOH. M.p. 170.5-171.5°.

*Chloride*:  $C_7H_2O_7N_3Cl$ . MW, 275.5. Plates from  $C_6H_6$ . M.p. 163° (158°). Spar. sol.  $Et_2O$ .

*Amide*:  $C_7H_4O_7N_4$ . MW, 256. Cryst. from  $Me_2CO$ - $C_6H_6$ -pet. ether. M.p. 264°.

*Methylamide*:  $C_8H_6O_7N_4$ . MW, 270. M.p. 285°.

*Dimethylamide*:  $C_9H_8O_7N_4$ . MW, 284. M.p. 144°.

*Anhydride*:  $C_{14}H_4O_{15}N_6$ . MW, 496. Needles. M.p. 270°.

Clarke, Hartman, *Organic Syntheses*, Collective Vol. I, 528.

Chang, Kao, *J. Chinese Chem. Soc.*, 1935, 3, 256.

Krauz, Turek, *Chimie et industrie*, 1926, Special No., 526.

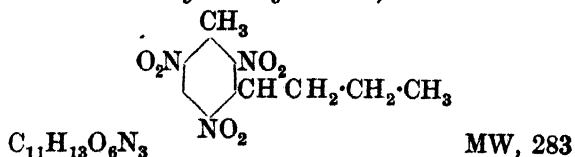
Lüttgen, D.R.P., 226,225, (*Chem. Zentr.*, 1910, II, 1174).

### 3 : 4 : 5-Trinitrobenzoic Acid.

Greenish-yellow needles +  $Et_2O$  from  $Et_2O$ . M.p. 168° decomp.

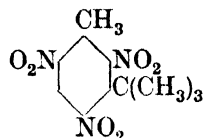
Körner, Contardi, *Atti accad. Lincei*, 1914, 23, II, 467.

2 : 4 : 6-Trinitro-3-*n*-butyltoluene (2 : 4 : 6-Trinitro-3-methyl-*n*-butylbenzene)



Fine needles from EtOH. M.p. 78.5°. Odourless. Turns pink, then yellowish-brown in light. de Capeller, *Helv. Chim. Acta*, 1928, 11, 168.

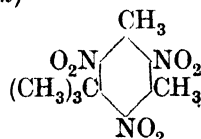
2 : 4 : 6 - Trinitro - 3 - *tert.* - butyltoluene (2 : 4 : 6-Trinitro-3-methyl-*tert.*-butylbenzene)



Yellowish needles from EtOH. M.p. 96-7°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ , pet. ether. Insol.  $H_2O$ . Strong odour of musk. Non-poisonous.

Baur, *Compt. rend.*, 1890, 111, 239.

2 : 4 : 6-Trinitro-5-*tert.*-butyl-*m*-xylene (*Xylene musk*)

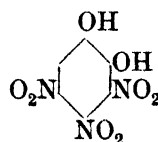


Needles from EtOH. M.p. 110°. Possesses strong musk-like odour.

Baur, *Ber.*, 1891, 24, 2841.

Fabr. de Thann et Mulhouse, D.R.P., 77,299.

### 3 : 4 : 5-Trinitrocatechol

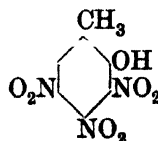


*Di-Me ether*: see 3 : 4 : 5-Trinitroveratrol.

*Di-Et ether*:  $C_{10}H_{11}O_8N_3$ . MW, 301. Needles. M.p. 122°.

Blanksma, *Rec. trav. chim.*, 1905, 24, 42.

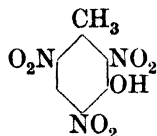
### 3 : 4 : 5-Trinitro-*o*-cresol



Orange-yellow prisms from  $Me_2CO$ . M.p. 102°. Very sol.  $CHCl_3$ . Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ , AcOEt. Spar. sol. cold  $H_2O$ .

*Me ether*:  $C_8H_7O_7N_3$ . MW, 257. Needles from EtOH.Aq. M.p. 111–12°. Sol. hot EtOH.Aq., AcOH.

Sommer, *J. prakt. Chem.*, 1903, **67**, 554.  
Nölting, Collin, *Ber.*, 1884, **17**, 270.

**2 : 4 : 6-Trinitro-*m*-cresol**

$C_7H_5O_7N_3$  MW, 243

Yellow needles from EtOH. M.p. 109–10°. Sol. 123 parts boiling and 446 parts cold  $H_2O$ . Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ .

*Me ether*:  $C_8H_7O_7N_3$ . MW, 257. Prisms from EtOH. M.p. 94°.

*Et ether*:  $C_9H_9O_7N_3$ . MW, 271. Leaflets from EtOH. M.p. 75°. Sol.  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Turns yellow in air.

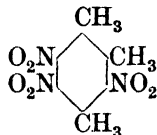
*Acetyl*: pale yellow plates from  $C_6H_6$ . M.p. 135°.

Datta, Varma, *J. Am. Chem. Soc.*, 1919, **41**, 2041.

Giua, *Gazz. chim. ital.*, 1919, **49**, ii, 164.

Beilstein, Kellner, *Ann.*, 1863, **128**, 165.

**Trinitro- $\psi$ -cumene** (3 : 5 : 6-Trinitro-1 : 2 : 4-trimethylbenzene)

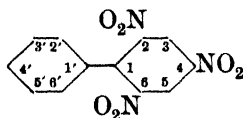


$C_9H_9O_6N_3$  MW, 255

Prisms. M.p. 185°. Very sol. boiling  $C_6H_6$ , toluene. Spar. sol. EtOH.

Schultz, *Ber.*, 1909, **42**, 3608.

Fittig, Laubinger, *Ann.*, 1869, **151**, 261.

**2 : 4 : 6-Trinitrodiphenyl**

$C_{12}H_7O_6N_3$  MW, 289

Pale yellowish needles from EtOH. M.p. 130°.

Gull, Turner, *J. Chem. Soc.*, 1929, 498.

**2 : 4 : 2'-Trinitrodiphenyl.**

Pale yellow prisms from AcOH. M.p. 150–1°.

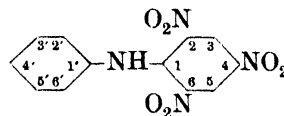
See previous reference.

**2 : 4 : 4'-Trinitrodiphenyl.**

Pale yellow cubes from AcOH or  $HNO_3$ . M.p. 176°.

See previous reference.

**2 : 4 : 6-Trinitrodiphenylamine** (*Picryl-aniline*, *N-phenylpicramide*)



$C_{12}H_9O_6N_4$  MW, 304

Yellow needles from EtOH. M.p. 179° (177°). Sol. warm AcOH,  $C_6H_6$ . Insol. ligroin.

Giua, Cherchi, *Gazz. chim. ital.*, 1919, **49**, ii, 157.

Ullmann, Nadai, *Ber.*, 1908, **41**, 1876.

Le Fèvre, *J. Chem. Soc.*, 1931, 813.

**2 : 4 : 2'-Trinitrodiphenylamine.**

Yellow prisms from EtOH or AcOH. M.p. 183–4°.

Juillard, *Bull. soc. chim.*, 1905, **33**, 1185.

**2 : 4 : 3'-Trinitrodiphenylamine.**

Pale brown plates or yellow needles from AcOH. M.p. 194–5° (190°). Mod. sol. hot AcOH. Spar. sol. hot EtOH. Conc.  $H_2SO_4$  → pale yellow sol.

v. der Kam, *Rec. trav. chim.*, 1926, **45**, 732.

Kym, Ringer, *Ber.*, 1915, **48**, 1681.

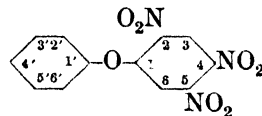
**2 : 4 : 4'-Trinitrodiphenylamine.**

Yellow needles from EtOH or AcOH. M.p. 189° (181°). Sol.  $Me_2CO$ . Spar. sol. AcOH, toluene.

See first reference above and also

Juillard, *Bull. soc. chim.*, 1905, **33**, 1182.

Wieland, Lecher, *Ann.*, 1912, **392**, 167.

**2 : 4 : 5-Trinitrodiphenyl Ether**

$C_{12}H_7O_7N_3$  MW, 305

Cryst. from EtOH. M.p. 106°. Sol. EtOH.

Westf.-Anhalt. Sprengstoff A.-G., D.R.P., 281,053, (*Chem. Zentr.*, 1915, I, 74).

**2 : 4 : 6-Trinitrodiphenyl Ether** (*Picric acid phenyl ether*, *phenyl picrate*).

Yellowish prisms from EtOH- $C_6H_6$ . M.p. 153°. Sol.  $C_6H_6$ . Mod. sol. EtOH,  $CHCl_3$ , AcOH. Spar. sol.  $Et_2O$ . Insol.  $H_2O$ , ligroin.

Jackson, Earle, *Am. Chem. J.*, 1903, **29**, 213.

**2 : 4 : 2'-Trinitrodiphenyl Ether.**

Yellow needles from EtOH-AcOH. M.p. 137.5°.

Raiford, Colbert, *J. Am. Chem. Soc.*, 1926, **48**, 2660.

**2 : 4 : 3'-Trinitrodiphenyl Ether.**

Yellow tablets from EtOH-AcOH. M.p. 136°.

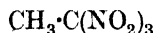
Raiford, Colbert, *J. Am. Chem. Soc.*, 1926, **48**, 2660.

Westf.-Anhalt. Sprengstoff A.-G., D.R.P., 281,053, (*Chem. Zentr.*, 1915, I, 74).

**2 : 4 : 4'-Trinitrodiphenyl Ether.**

Yellow tablets from EtOH-AcOH. M.p. 116° (100°).

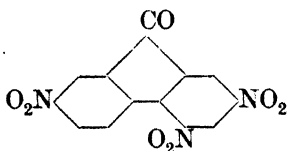
See first reference above.

**1 : 1 : 1-Trinitroethane**

$\text{C}_2\text{H}_3\text{O}_6\text{N}_3$  MW, 167

Cryst. M.p. 56°. Sol. usual org. solvents. Spar. sol.  $\text{H}_2\text{O}$ . Heat of comb.  $\text{C}_v$  1777 Cal. Very volatile.

Hantzsch, Rinckenberger, *Ber.*, 1899, **32**, 637.

**2 : 4 : 7-Trinitrofluorenone**

$\text{C}_{13}\text{H}_5\text{O}_7\text{N}_3$  MW, 315

Pale yellow needles from AcOH or  $\text{C}_6\text{H}_6$ . M.p. 176°. Very sol.  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .

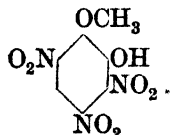
*Oxime*: needles from EtOH. M.p. 260° decomp.

*Semicarbazone*: pale yellow. M.p. 299° decomp.

*Phenylhydrazone*: violet leaflets. M.p. 276° decomp.

Bell, *J. Chem. Soc.*, 1928, 1990.

Schmidt, Bauer, *Ber.*, 1905, **38**, 3760.

**3 : 4 : 6-Trinitroguaiacol**

$\text{C}_7\text{H}_5\text{O}_8\text{N}_3$  MW, 259

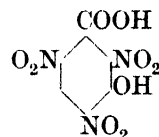
Yellow prisms from  $\text{CHCl}_3$ . M.p. 129° decomp.

*Me ether*: see 3 : 4 : 6-Trinitroveratrol.

Pollecioff, Robinson, *J. Chem. Soc.*, 1918, **113**, 653.

**1 : 3 : 5-Trinitrohexahydro-1 : 3 : 5-triazine.**

See Hexogen.

**2 : 4 : 6-Trinitro-*m*-hydroxybenzoic Acid**

$\text{C}_7\text{H}_3\text{O}_9\text{N}_3$  MW, 273

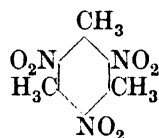
Plates and prisms +  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. about 105°. Sublimes. Blackens at 200°. Sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Bitter taste.

*Nitrile*:  $\text{C}_7\text{H}_2\text{O}_7\text{N}_4$ . MW, 254. Yellowish needles or leaflets from dil.  $\text{HNO}_3$ . M.p. 131-2°.

Schardinger, *Ber.*, 1875, **8**, 1487.

Borsche, Gahrtz, *Ber.*, 1906, **39**, 3365.

Beilstein, Geitner, *Ann.*, 1866, **139**, 11.

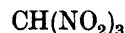
**Trinitromesitylene**

$\text{C}_9\text{H}_9\text{O}_6\text{N}_3$  MW, 255

Needles from EtOH, prisms from  $\text{Me}_2\text{CO}$ . M.p. 235° (232°). Mod. sol.  $\text{Me}_2\text{CO}$ . Spar. sol. hot EtOH,  $\text{Et}_2\text{O}$ . Explosive.

Fittig, *Ann.*, 1867, **141**, 134.

Blanksma, *Rec. trav. chim.*, 1902, **21**, 336.

**Trinitromethane (Nitroform)**

$\text{CHO}_6\text{N}_3$  MW, 151

Colourless cryst. M.p. 15°. B.p. 45-7°/22 mm.  $D_4^{25}$  1.5967.  $n_D^{25}$  1.44511. Sol.  $\text{H}_2\text{O}$  with intense yellow col. Explodes on rapid heating. Forms metallic salts.

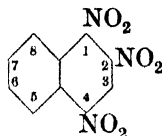
*NH<sub>4</sub> salt*: yellow needles from  $\text{H}_2\text{O}$ . Decomp. about 200°.

*K salt*: yellow cryst. from  $\text{H}_2\text{O}$ . Explodes at 97-9°. Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ .

Chattaway, Harrison, *J. Chem. Soc.*, 1916, **109**, 171.

Hantzsch, Rinckenberger, *Ber.*, 1899, **32**, 631.

## 1 : 2 : 4-Trinitronaphthalene

 $C_{10}H_5O_6N_3$ 

MW, 263

Yellow needles from AcOH. M.p. 258°.

Contardi, Mor, *Chem. Abstracts*, 1925, 19, 827.

## 1 : 2 : 5-Trinitronaphthalene.

Needles from EtOH. M.p. 112–13°.

Will, *Ber.*, 1895, 28, 377.

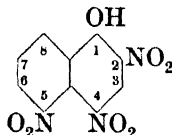
## 1 : 3 : 5-Trinitronaphthalene.

Rhombhedra from  $CHCl_3$ . M.p. 122°. Sol. EtOH, AcOH. Mod. sol.  $CHCl_3$ .de Aguiar, *Ber.*, 1872, 5, 897.

## 1 : 3 : 8-Trinitronaphthalene.

Cryst. from EtOH,  $CHCl_3$ , or AcOH. M.p. 218°. Spar. sol.  $Et_2O$ ,  $CHCl_3$ .Friedländer, *Ber.*, 1899, 32, 3531.Kalle, D.R.P., 117,368, (*Chem. Zentr.*, 1901, I, 347).Rindl, *J. Chem. Soc.*, 1913, 103, 1914.

## 1 : 4 : 5-Trinitronaphthalene.

Pale yellow leaflets from  $HNO_3$ . M.p. 154°. Sol. 95 parts  $C_6H_6$ , 156 parts  $CHCl_3$ , 260 parts  $Et_2O$ , 894 parts 90% EtOH, 4017 parts  $CS_2$ , 20,193 parts ligroin, at 18–5°.de Aguiar, *Ber.*, 1872, 5, 904.2 : 4 : 5-Trinitro-1-naphthol (*Naphthopipric acid*) $C_{10}H_5O_7N_3$ 

MW, 279

Yellow leaflets or prisms from AcOH. M.p. 189–5°. Sol. 364 parts cold AcOH. Spar. sol. EtOH,  $Et_2O$ , AcOEt,  $C_6H_6$ , xylene, hot  $H_2O$ .*Me ether*:  $C_{11}H_7O_7N_3$ . MW, 293. Yellow leaflets from AcOH. M.p. 150–5–151–5° (128°). Spar. sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ .*Et ether*:  $C_{12}H_9O_7N_3$ . MW, 307. Yellow needles from EtOH. M.p. 149–50°. Very sol. AcOH. Spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .Diehl, Merz, *Ber.*, 1878, 11, 1662.Kehrmann, Steiner, *Ber.*, 1900, 33, 3281.Staedel, *Ann.*, 1883, 217, 170.Rindl, *J. Chem. Soc.*, 1913, 103, 1913.

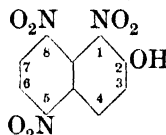
## 2 : 4 : 7-Trinitro-1-naphthol.

Yellow prisms from  $C_6H_6$  or AcOH. M.p. 145°.Kehrmann, Haberkant, *Ber.*, 1898, 21, 2420.Kehrmann, Steiner, *Ber.*, 1900, 33, 3285.

## 2 : 4 : 8-Trinitro-1-naphthol.

Yellow prisms. M.p. 175°. Spar. sol. cold  $H_2O$ .Graebe, *Ber.*, 1899, 32, 2879.Rindl, *J. Chem. Soc.*, 1913, 103, 1916.

## 1 : 5 : 8-Trinitro-2-naphthol

 $C_{10}H_5O_7N_3$ 

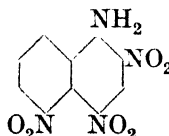
MW, 279

*Me ether*:  $C_{11}H_7O_7N_3$ . MW, 293. Yellow needles from AcOH. M.p. 191°.Will, *Ber.*, 1895, 28, 372.

## 1 : 6 : 8-Trinitro-2-naphthol.

*Me ether*: needles from AcOH. M.p. 213°. Insol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .*Et ether*:  $C_{12}H_9O_7N_3$ . MW, 307. Pale yellow needles from AcOH. M.p. 186°. Spar. sol. EtOH,  $CHCl_3$ ,  $C_6H_6$ .Staedel, *Ann.*, 1883, 217, 171.

## 2 : 4 : 5-Trinitro-1-naphthylamine

 $C_{10}H_6O_6N_4$ 

MW, 278

Yellow needles from  $Me_2CO$ . M.p. 315–17° (decomp. at 310°). Mod. sol.  $Me_2CO$ ,  $Ac_2O$ . Spar. sol. most other solvents.*N-Me*:  $C_{11}H_8O_6N_4$ . MW, 292. Light brown leaflets from AcOEt. M.p. 206°. Sol.  $Me_2CO$  warm AcOH. Mod. sol. AcOEt,  $CHCl_3$ . Spar. sol. EtOH,  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ , pet. ether.*N-Di-Me*:  $C_{12}H_{10}O_6N_4$ . MW, 306. Orange leaflets from AcOH. M.p. 194–5–195–5°.*N-Et*:  $C_{12}H_{10}O_6N_4$ . MW, 306. Yellowish-brown needles from EtOH. M.p. 160°. Sol.  $Me_2CO$ . Mod. sol. AcOH,  $CHCl_3$ ,  $C_6H_6$ , warm EtOH. Spar. sol.  $Et_2O$ ,  $CS_2$ , pet. ether.*N-Propyl*:  $C_{13}H_{12}O_6N_4$ . MW, 320. Yellow needles from AcOH. M.p. 139°.*N-Butyl*:  $C_{14}H_{14}O_6N_4$ . MW, 334. Yellow needles from AcOH. M.p. 121°.



N-Amyl:  $C_{15}H_{16}O_6N_4$ . MW, 348. Yellow needles. M.p. 144-5°.

N-Phenyl:  $C_{16}H_{10}O_6N_4$ . MW, 354. Plates from AcOH. M.p. 218.5°. Mod. sol. AcOH,  $C_6H_6$ . Insol. EtOH,  $Et_2O$ , pet. ether.

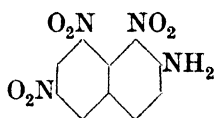
N-Acetyl: cryst. from AcOH. Decomp. at 275°. Sol.  $Ac_2O$ . Mod. sol. AcOH.

Talen, *Rec. trav. chim.*, 1928, **47**, 355.

Groeneveld, *Rec. trav. chim.*, 1931, **50**, 692.

Rindl, *J. Chem. Soc.*, 1913, **103**, 1915.

### 1 : 6 : 8-Trinitro-2-naphthylamine



$C_{10}H_6O_6N_4$  MW, 278

M.p. 300° decomp. Sol.  $Me_2CO$ , AcOH,  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Insol.  $CS_2$ , pet. ether.

N-Me: m.p. 257° decomp.

N-Di-Me: pale yellow needles. M.p. 226°.

N-Et: orange needles. M.p. 216°.

N-Propyl: m.p. 186°. Acetyl: m.p. 179-80°.

N-Isopropyl: m.p. 209°.

N-Butyl: yellow needles. M.p. 156°.

N-Isobutyl: m.p. 179°.

N-Amyl: m.p. 181°.

N-Isoamyl: yellow needles. M.p. 164°.

N-Acetyl: pale yellow cryst. from AcOH. M.p. 239-40°. Sol. AcOH,  $Me_2CO$ . Mod. sol. EtOH, AcOEt, pet. ether.

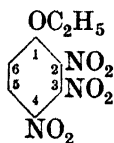
v. der Kam, *Rec. trav. chim.*, 1926, **45**, 727.

Staedel, *Ann.*, 1883, **217**, 174.

### Trinitrophenetidine.

See under Trinitroaminophenol.

### 2 : 3 : 4-Trinitrophenetole



$C_8H_7O_7N_3$  MW, 257

Cryst. from EtOH. M.p. 117°.

Blanksma, *Rec. trav. chim.*, 1908, **27**, 51.

### 2 : 3 : 5-Trinitrophenetole.

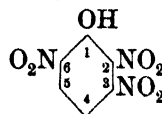
Pale yellow cryst. M.p. 80°.

Blanksma, *Rec. trav. chim.*, 1905, **24**, 41.

### 2 : 4 : 6-Trinitrophenetole.

See under Picric Acid.

### 2 : 3 : 6-Trinitrophenol



$C_6H_3O_7N_3$  MW, 229

Yellow needles from  $H_2O$ . M.p. 119°. Sol. EtOH, AcOH,  $C_6H_6$ .

Henriques, *Ann.*, 1882, **215**, 325.

See also Reverdin, Meldola, *J. prakt. Chem.*, 1913, **88**, 796.

### 2 : 4 : 5-Trinitrophenol.

Needles from  $H_2O$  or EtOH.Aq. M.p. 96°. Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Spar. sol. cold  $H_2O$ . Very bitter taste.

Me ether: see 2 : 4 : 5-Trinitroanisole.

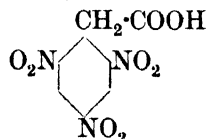
Phenyl ether: see 2 : 4 : 5-Trinitrodiphenyl Ether.

Henriques, *Ann.*, 1882, **215**, 329.

### 2 : 4 : 6-Trinitrophenol.

See Picric Acid.

### 2 : 4 : 6-Trinitrophenylacetic Acid

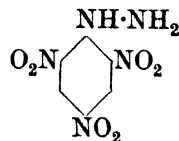


$C_8H_5O_8N_3$  MW, 271

Needles from  $C_6H_6$ . M.p. 161°. Heat with  $H_2O$  or EtOH  $\rightarrow$  2 : 4 : 6-trinitrotoluene.

Jackson, Phinney, *Am. Chem. J.*, 1899, **21**, 430.

### 2 : 4 : 6-Trinitrophenylhydrazine (Picrylhydrazine)



$C_8H_5O_6N_5$  MW, 243

Red plates from EtOH. M.p. 186° (175°). Sol. AcOH. Mod. sol. EtOH. Spar. sol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Reduces Fehling's and  $NH_3 \cdot AgNO_3$ .

Acetyl: m.p. 223° (214° decomp.).

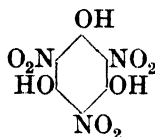
Giua, Cherchi, *Gazz. chim. ital.*, 1919, **49**, ii, 152.

Curtius, Dedichen, *J. prakt. Chem.*, 1894, **50**, 271.

### 2 : 4 : 6-Trinitrophenylnitramine.

See Picrylnitramine.

## Trinitrophloroglucinol

 $C_6H_3O_9N_3$ 

MW, 261

Yellow needles +  $H_2O$  from  $H_2O$ . M.p. anhyd.  $147^\circ$  decomp. Sol. hot  $H_2O$ , EtOH,  $Et_2O$ . Explodes above m.p. Sublimes.

*Tri-Et ether*:  $C_{12}H_{15}O_9N_3$ . MW, 345. Plates from EtOH. M.p.  $119-20^\circ$ . Very sol.  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Sol. hot EtOH, AcOH. Mod. sol.  $CS_2$ . Spar. sol. ligroin.

*Triphenyl ether*:  $C_{24}H_{15}O_9N_3$ . MW, 489. Needles from  $C_6H_6$ -EtOH. M.p.  $175^\circ$ . Very sol.  $Me_2CO$ ,  $CHCl_3$ . Sol. AcOH,  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ ,  $CS_2$ , ligroin.

*Tribenzyl ether*:  $C_{27}H_{21}O_9N_3$ . MW, 531. Needles from EtOH- $C_6H_6$ . M.p.  $171^\circ$ .

Freundenberg, Fikentscher, Wenner, *Ann.*, 1925, 442, 322.

Jackson, Warren, *Am. Chem. J.*, 1893, 15, 611.

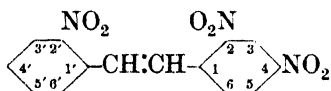
Jackson, Smith, *Am. Chem. J.*, 1904, 32, 173.

Blanksma, *Rec. trav. chim.*, 1908, 27, 35.

## 2 : 4 : 6-Trinitroresorcinol.

See Styphnic Acid.

## 2 : 4 : 2'-Trinitrostilbene

 $C_{14}H_9O_6N_3$ 

MW, 315

Greenish-yellow cryst. from AcOH. M.p.  $194-5^\circ$ .

Thiele, Escales, *Ber.*, 1901, 34, 2847.

## 2 : 4 : 3'-Trinitrostilbene.

Yellow needles from AcOH. M.p.  $183-4^\circ$ . Spar. sol. boiling EtOH.

See previous reference.

## 2 : 4 : 4'-Trinitrostilbene.

Dark orange needles from AcOH. M.p.  $240^\circ$  ( $234-5^\circ$ ). Sol. about 70 parts boiling AcOH.

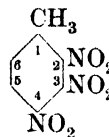
Nisbet, *J. Chem. Soc.*, 1927, 2082.

Thiele, Escales, *Ber.*, 1901, 34, 2846.

Bayer, D.R.P., 124,681, (*Chem. Zentr.*, 1901, II, 1029).

Dict. of Org. Comp.—III

## 2 : 3 : 4-Trinitrotoluene

 $C_7H_5O_6N_3$ 

MW, 227

Prisms from  $Me_2CO$ . M.p.  $112^\circ$ . Sol.  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Mod. sol. boiling EtOH. Explodes at  $290-310^\circ$ .

Gornall, Robinson, *J. Chem. Soc.*, 1926, 1981.

Will, *Ber.*, 1914, 47, 710.

## 2 : 3 : 5-Trinitrotoluene.

Yellowish prisms from EtOH- $Et_2O$ . M.p.  $97-5^\circ$ .

Körner, Contardi, *Atti accad. Lincei*, 1915, 24, I, 891.

## 2 : 3 : 6-Trinitrotoluene.

Needles from EtOH. M.p.  $111^\circ$ . Sol. 9 parts boiling EtOH.

Körner, Contardi, *Atti accad. Lincei*, 1916, 25, II, 345.

Brady, Taylor, *J. Chem. Soc.*, 1920, 117, 876.

## 2 : 4 : 5-Trinitrotoluene.

Yellowish plates from  $Me_2CO$ . M.p.  $104^\circ$ . Sol.  $Et_2O$ ,  $Me_2CO$ ,  $C_6H_6$ . Mod. sol. hot EtOH, AcOH. Explodes at  $290-310^\circ$ .

Hepp, *Ann.*, 1882, 215, 366.

Will, *Ber.*, 1914, 47, 710.

## 2 : 4 : 6-Trinitrotoluene.

Rhomboheda from EtOH. M.p.  $80-8^\circ$ . Very sol.  $C_6H_6$ . Mod. sol.  $Et_2O$ . Sol. 700 parts boiling  $H_2O$ . This compound is the explosive known as T.N.T., but the commercial product usually contains small quantities of other nitrotoluenes according to the method of manufacture.

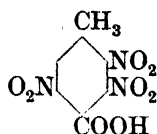
Copisarow, *Chem. News*, 1916, 113, 37; 1915, 112, 247.

Jackson, Phinney, *Am. Chem. J.*, 1899, 21, 431.

## 3 : 4 : 5-Trinitrotoluene.

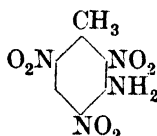
Greenish-yellow prisms or plates from EtOH. M.p.  $137-5^\circ$ . Sol. 100 parts 95% EtOH at  $15^\circ$ .

Körner, Contardi, *Atti accad. Lincei*, 1914, 23, II, 466.

2 : 3 : 5-Trinitro-*p*-toluic Acid $C_8H_5O_6N_3$ 

MW, 271

White plates. M.p. 230–1°.

*Me ester*:  $C_9H_7O_6N_3$ . MW, 285. Needles from MeOH. M.p. 114–15°.*Et ester*:  $C_{10}H_9O_6N_3$ . MW, 299. Prisms from EtOH. M.p. 87–8°.Giua, *Gazz. chim. ital.*, 1922, 52, i, 183.2 : 4 : 6-Trinitro-*m*-toluidine $C_7H_6O_6N_4$ 

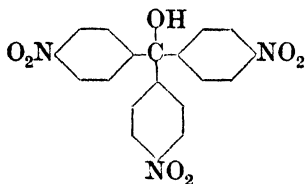
MW, 242

Yellow prismatic leaflets from EtOH. M.p. 138° (136°). Sol. EtOH, Et<sub>2</sub>O.*N-Me*:  $C_8H_8O_6N_4$ . MW, 256. Yellow cryst. M.p. 138°.*N-Et*:  $C_9H_{10}O_6N_4$ . MW, 270. Yellow cryst. M.p. 98°.Blanksma, *Rec. trav. chim.*, 1902, 21, 332.Reverdin, Dresel, Delétra, *Ber.*, 1904, 37, 2095.

## Trinitrotrimethylenetriamine.

See Hexogen.

## 4 : 4' : 4''-Trinitrotriphenylcarbinol

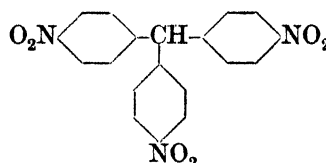
 $C_{19}H_{13}O_7N_3$ 

MW, 395

Exists in two modifications.

(i) Prisms from AcOH or  $C_6H_6$ . M.p. 189°.(ii) Rhombohedra from  $C_6H_6$  or MeOH. M.p. 167°.Sol. AcOH,  $C_6H_6$ . Spar. sol. hot EtOH, Et<sub>2</sub>O, CS<sub>2</sub>. Heat of comb.  $C_6$  2218 Cal.Fischer, Schmidt, *Chem. Zentr.*, 1904, I, 460.Fischer, Fischer, *Ber.*, 1904, 37, 3355.

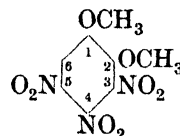
## 4 : 4' : 4''-Trinitrotriphenylmethane

 $C_{19}H_{13}O_6N_3$ 

MW, 379

Cryst. from  $C_6H_6$ . M.p. 212–5°. Spar. sol. AcOH,  $C_6H_6$ . Sol. alc. KOH with intense violet-blue col. Heat of comb.  $C_6$  2272.8 Cal.Montagne, *Rec. trav. chim.*, 1905, 24, 125.Hantzsch, Hein, *Ber.*, 1919, 52, 495.

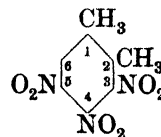
## 3 : 4 : 5-Trinitroveratrol

 $C_8H_7O_8N_3$ 

MW, 273

Prisms from EtOH. M.p. 145°. Sol. hot EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.Vermeulen, *Rec. trav. chim.*, 1929, 48, 969.Klemenc, *Monatsh.*, 1912, 33, 389.Blanksma, *Rec. trav. chim.*, 1904, 23, 114.

## 3 : 4 : 6-Trinitroveratrol.

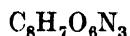
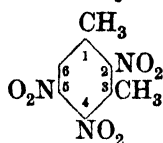
Pale yellow needles from EtOH. M.p. 174°. Sol. Me<sub>2</sub>CO, AcOEt. Spar. sol. EtOH, AcOH, CHCl<sub>3</sub>. Decomp. slowly by cold NaOH and boiling Na<sub>2</sub>CO<sub>3</sub>-Aq.Pollecöff, Robinson, *J. Chem. Soc.*, 1918, 113, 655.3 : 4 : 5-Trinitro-*o*-xylene $C_8H_7O_6N_3$ 

MW, 241

Needles from EtOH. M.p. 115°. Sol. Me<sub>2</sub>CO, AcOEt, CHCl<sub>3</sub>,  $C_6H_6$ . Mod. sol. EtOH.Crossley, Renouf, *J. Chem. Soc.*, 1909, 95, 211.3 : 4 : 6-Trinitro-*o*-xylene.

Yellowish needles from EtOH. M.p. 72°. Sol. most org. solvents except EtOH, pet. ether. Turns yellow in air.

See previous reference.

2 : 4 : 5-Trinitro-*m*-xylene

MW, 241

Yellow cryst. from EtOH. M.p. 90°.  $D^{14}_{1.553}$ . Sol. 50 parts EtOH at 25°. Sol.  $HNO_3$ .

Blanksma, *Rec. trav. chim.*, 1909, **28**, 95; 1906, **25**, 167.

2 : 4 : 6-Trinitro-*m*-xylene.

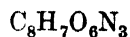
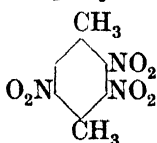
Pale yellow prisms or leaflets from  $C_6H_6$ -EtOH. M.p. 182°.  $D^{19}_{1.604}$ . Sol. 2500 parts EtOH at 20°. Spar. sol. warm  $HNO_3$ .

Blanksma, *Rec. trav. chim.*, 1906, **25**, 178.  
Beilstein, Luhmann, *Ann.*, 1867, **144**, 274.

4 : 5 : 6-Trinitro-*m*-xylene.

Prisms from EtOH. M.p. 125°.  $D^{19}_{1.494}$ . Sol. 80 parts EtOH at 20°. Sol. warm  $HNO_3$ .

Blanksma, *Rec. trav. chim.*, 1906, **25**, 168.

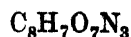
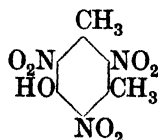
2 : 3 : 5-Trinitro-*p*-xylene

MW, 241

Needles from EtOH or leaflets from EtOH- $C_6H_6$ . M.p. 139-40° (137°).  $D^{19}_{1.59}$ . Turns yellow in air.

Fittig, Ahrens, Mattheides, *Ann.*, 1868, **147**, 23.

Giua, *Gazz. chim. ital.*, 1919, **49**, ii, 149 (Note).

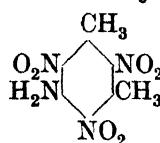
2 : 4 : 6-Trinitro-*m*-5-xylenol (*Xylopicric acid*)

MW, 257

Needles from dil.  $HNO_3$ . M.p. 108° (104°). Spar. sol.  $H_2O$  with yellow col.

*Me ether*:  $C_9H_9O_7N_3$ . MW, 271. Needles from EtOH. M.p. 127°. Sol. AcOH, hot EtOH. Insol.  $H_2O$ .

Blanksma, *Rec. trav. chim.*, 1902, **21**, 329.  
Knecht, Hibbert, *Ber.*, 1904, **37**, 3477.

2 : 4 : 6-Trinitro-*m*-5-xyldine

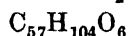
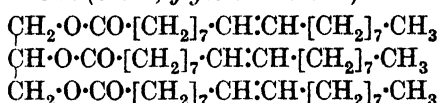
MW, 256

Yellow cryst. M.p. 206°.

*N-Me*:  $C_9H_{10}O_6N_4$ . MW, 270. Yellow cryst. M.p. 165°.

*N-Et*:  $C_{10}H_{12}O_6N_4$ . MW, 284. M.p. 122°.

Blanksma, *Rec. trav. chim.*, 1906, **25**, 374; 1902, **21**, 331.

Triolein (*Olein, glycerol trioleate*)

MW, 884

Found in olive oil, butter fat, and other vegetable oils. F.p. -4°. B.p. 235-40°/18 mm.  $D^{20}_4$  0.8992.  $n^{20}_D$  1.4561. Very sol.  $Et_2O$ . Spar. sol. EtOH. Tasteless. Odourless.

Amberger, Bromig, *Biochem. Z.*, 1922, **130**, 252.

Bellucci, *Gazz. chim. ital.*, 1912, **42**, ii, 291.

Bournot, *Biochem. Z.*, 1914, **65**, 156.

## Trional.

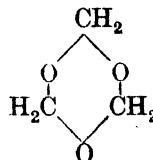
See Methylsulphonal.

## Triorsellinic Acid.

See Gyrophoric Acid.

## 1 : 3 : 5-Trioxan.

See Trioxymethylene.

Trioxymethylene (*Metaformaldehyde, 1:3:5-trioxan*)

MW, 90

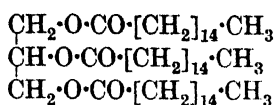
Cryst. from  $Et_2O$ . M.p. 64° (61°). B.p. 114.5°/759 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $CCl_4$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol. pet. ether. Sol. 5 parts  $H_2O$  at 25°. Sublimes at 46° undecomp. in presence of traces of moisture.

*Diacetyl*: b.p. 113-15°/2 mm. Misc. with  $Et_2O$ .

Staudinger, Lüthy, *Helv. Chim. Acta*, 1925, **8**, 51, 65.

Hammick, Boeree, *J. Chem. Soc.*, 1922, **121**, 2738.

**Tripalmitin** (*Palmitin, glycerol tripalmitate*)



$\text{C}_{51}\text{H}_{98}\text{O}_6$  MW, 806

Needles from  $\text{Et}_2\text{O}$ . M.p.  $65.5^\circ$ .  $D_4^{20}$  0.8752.  $n_D^{20}$  1.43807. Very sol.  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ .

Bellucci, *Gazz. chim. ital.*, 1912, **42**, ii, 290.

Stephenson, *Biochem. J.*, 1913, **7**, 432.

Averill, Roche, King, *J. Am. Chem. Soc.*, 1929, **51**, 870.

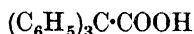
**Tri-perinaphthylenebenzene.**

See Decacyclene.

**Tripetroselin.**

See under Petroselic Acid.

**Triphenylacetic Acid** (*Triphenylmethane- $\alpha$ -carboxylic acid*)



$\text{C}_{20}\text{H}_{16}\text{O}_2$  MW, 288

Prisms from  $\text{EtOH}$ . M.p.  $267^\circ$ . Mod. sol.  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{AcOH}$ , ligroin. Spar. sol.  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Very weak acid. Above m.p. part. decomp.  $\rightarrow \text{CO}_2$  + triphenylmethane. Warm conc.  $\text{H}_2\text{SO}_4 \rightarrow \text{CO}$  + triphenylcarbinol.

*Me ester*:  $\text{C}_{21}\text{H}_{18}\text{O}_2$ . MW, 302. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $186^\circ$ . Sol.  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{MeOH}$ ,  $\text{Et}_2\text{O}$ , ligroin.

*Et ester*:  $\text{C}_{22}\text{H}_{20}\text{O}_2$ . MW, 316. M.p.  $120-1^\circ$ .

*Propyl ester*:  $\text{C}_{23}\text{H}_{22}\text{O}_2$ . MW, 330. M.p.  $98-9^\circ$ .

*Isopropyl ester*: m.p.  $84^\circ$ .

*Butyl ester*:  $\text{C}_{24}\text{H}_{24}\text{O}_2$ . MW, 344. M.p.  $99-99.6^\circ$ .

*sec.-n-Butyl ester*: m.p.  $101-2^\circ$ .

*Isobutyl ester*: m.p.  $88-9^\circ$ .

*n-Amyl ester*:  $\text{C}_{25}\text{H}_{26}\text{O}_2$ . MW, 358. M.p.  $76^\circ$ .

*Isoamyl ester*: needles. M.p.  $78.5-79.5^\circ$ .

*Phenyl ester*:  $\text{C}_{26}\text{H}_{20}\text{O}_2$ . MW, 364. Needles from  $\text{C}_6\text{H}_6$ . M.p.  $124.5-125^\circ$ .

*Benzyl ester*: m.p.  $99.5^\circ$ .

*Chloride*:  $\text{C}_{20}\text{H}_{15}\text{OCl}$ . MW, 306.5. Prisms from  $\text{C}_6\text{H}_6$  or ligroin. M.p.  $128-9^\circ$  decomp.

*Amide*:  $\text{C}_{20}\text{H}_{17}\text{ON}$ . MW, 287. Prisms from toluene. M.p.  $246-7^\circ$  ( $238^\circ$ ). Very spar. sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .

*Nitrile*:  $\alpha$ -cyanotriphenylmethane.  $\text{C}_{20}\text{H}_{15}\text{N}$ . MW, 269. Plates from  $\text{Me}_2\text{CO}$ . Aq. M.p.  $127-8^\circ$ .

*Anhydride*:  $\text{C}_{40}\text{H}_{30}\text{O}_3$ . MW, 558. Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $163^\circ$ .

*Anilide*: cryst. from  $\text{AcOH}$ . M.p.  $173.5-174.5^\circ$  ( $167-8^\circ$ ). *N-Benzoyl*: needles from  $\text{C}_6\text{H}_6$ . M.p.  $185-6^\circ$ .

Norris, Cresswell, *J. Am. Chem. Soc.*, 1934, **56**, 423.

Schlenk, Marcus, *Ber.*, 1914, **47**, 1666.

Elbs, Tölle, *J. prakt. Chem.*, 1885, **32**, 624.

Bistrzycki, Mauron, *Ber.*, 1907, **40**, 4062.

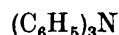
Schmidlin, Hodgson, *Ber.*, 1908, **41**, 441.

Fischer, Fischer, *Ann.*, 1878, **194**, 260.

**Triphenylacrylic Acid.**

See  $\alpha$ :  $\beta$ -Diphenylcinnamic Acid.

**Triphenylamine**



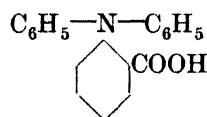
$\text{C}_{18}\text{H}_{15}\text{N}$  MW, 245

Cryst. from  $\text{AcOEt}$ . M.p.  $127^\circ$ . B.p.  $365^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{EtOH}$ . Does not combine with picric acid or alkyl iodides. *Hydrochloride*: m.p.  $214^\circ$ .

Hager, *Organic Syntheses*, Collective Vol. I, 529.

Piccard, Kharasch, *J. Am. Chem. Soc.*, 1918, **40**, 1077.

**Triphenylamine-2-carboxylic Acid** (*Diphenylanthranilic acid*)

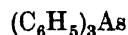


$\text{C}_{19}\text{H}_{15}\text{O}_2\text{N}$  MW, 289

Yellow feathery cryst. from  $\text{AcOH}$ . M.p.  $208^\circ$ . Heat above m.p.  $\rightarrow$  triphenylamine. Sol. conc.  $\text{H}_2\text{SO}_4$  with blue col.

Goldberg, Nimerovsky, *Ber.*, 1907, **40**, 2449.

**Triphenylarsine** (*Arsenic triphenyl*)



$\text{C}_{18}\text{H}_{15}\text{As}$  MW, 306

Needles from  $\text{EtOH}$ . Aq. M.p.  $60.5^\circ$ . B.p. above  $360^\circ$  in  $\text{CO}_2$ . Very sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. cold  $\text{EtOH}$ . Insol.  $\text{H}_2\text{O}$ .

$\text{B}_2\text{H}_2\text{PtCl}_6$ : yellow leaflets from  $\text{CHCl}_3$ . M.p.  $285^\circ$ .

*Hydroxymethylate*: cryst. from  $\text{EtOH}$ . Aq. M.p.  $125-6^\circ$ .

*Methochloride*: needles. M.p.  $121^\circ$ . Very sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*Methiodide*: yellow leaflets from  $\text{EtOH}$ . M.p.  $176^\circ$ .

*Ethiodide*: needles from EtOH-Et<sub>2</sub>O. M.p. 158°.

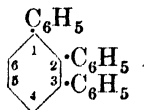
Pope, Turner, *J. Chem. Soc.*, 1920, 117, 1447.

Hilpert, Herrmann, *Ber.*, 1913, 46, 2223.

Philips, *Ber.*, 1886, 19, 1031.

Michaelis, *Ann.*, 1902, 321, 160.

### 1 : 2 : 3-Triphenylbenzene



C<sub>24</sub>H<sub>18</sub>

MW, 306

M.p. 150-5°.

Knoevenagel, Vieth, *Ann.*, 1894, 281, 72.

### 1 : 3 : 5-Triphenylbenzene.

Needles from AcOH. M.p. 172°. Distills undecomp. Sol. C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, Et<sub>2</sub>O. CrO<sub>3</sub> in AcOH → benzoic acid. Causes malignant growths in mice.

Odell, Hines, *J. Am. Chem. Soc.*, 1913, 35, 82.

Reddellien, *Ber.*, 1913, 46, 2716.

Knoll, D.R.P., 250,236, (*Chem. Zentr.*, 1912, II, 1084).

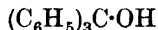
### Triphenylbenzoylmethane.

See Benzpinacolin.

### Triphenyl borate.

See under Phenol.

**Triphenylcarbinol** (*α*-Hydroxytriphenylmethane, tritanol)



C<sub>18</sub>H<sub>16</sub>O

MW, 260

Plates from EtOH. M.p. 164-5°. B.p. 380°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Heat of comb. C<sub>p</sub> 2342.0 Cal., C<sub>v</sub> 2340.0 Cal. H (+ Ni) at 400°. HI in AcOH, or hot H·COOH → triphenylmethane.

*Me ether*: C<sub>20</sub>H<sub>18</sub>O. MW, 274. Plates from MeOH. M.p. 83-4°.

*Et ether*: C<sub>21</sub>H<sub>20</sub>O. MW, 288. Cryst. from EtOH. M.p. 84-5°.

*Propyl ether*: C<sub>22</sub>H<sub>22</sub>O. MW, 302. Cryst. from EtOH-Et<sub>2</sub>O. M.p. 56°.

*Isopropyl ether*: m.p. 113°.

*Phenyl ether*: C<sub>25</sub>H<sub>20</sub>O. MW, 336. Leaflets or prisms from Et<sub>2</sub>O-pet. ether. M.p. 103°.

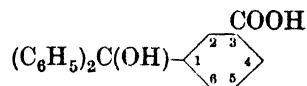
*Benzyl ether*: plates from EtOH. M.p. 106-7°.

*Acetyl*: triphenylmethyl acetate. Cryst. from AcOEt-ligroin. M.p. 87-8°.

Morton, Stevens, *J. Am. Chem. Soc.*, 1931, 53, 4030.

Stadnikow, *Ber.*, 1924, 57, 6.

### Triphenylcarbinol-3-carboxylic Acid (*α*-Hydroxytriphenylmethane-3-carboxylic acid)



C<sub>20</sub>H<sub>16</sub>O<sub>3</sub>

MW, 304

Cryst. from EtOH. M.p. 166-7°. Sol. conc. H<sub>2</sub>SO<sub>4</sub> with yellow col.

Bistrzycki, Gyr, *Ber.*, 1904, 37, 3698.

### Triphenylcarbinol-4-carboxylic Acid (*α*-Hydroxytriphenylmethane-4-carboxylic acid).

Needles from AcOH. M.p. 203.5° (200°). Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O.

*Me ester*: C<sub>21</sub>H<sub>17</sub>O<sub>3</sub>. MW, 318. Cryst. from MeOH. M.p. 119°.

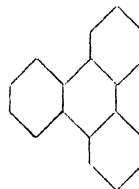
Staudinger, Clar, *Ber.*, 1911, 44, 1631.

Bistrzycki, Gyr, *Ber.*, 1904, 37, 657.

### Triphenyldihydroglyoxaline.

See Amarin.

### Triphenylene (9 : 10 - Benzphenanthrene, 1 : 2 : 3 : 4-dibenznaphthalene)



C<sub>18</sub>H<sub>12</sub>

MW, 228

Occurs to a small extent in coal-tar. Needles from EtOH. M.p. 198°. Sublimes. Sol. CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Mod. sol. EtOH, AcOH. Sols exhibit weak blue fluor. HNO<sub>3</sub> at 150° → mellitic acid.

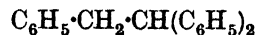
Mannich, *Ber.*, 1907, 40, 160.

Schmidt, Schultz, *Ann.*, 1880, 203, 135.

### 1 : 1 : 1-Triphenylethane.

See *α*-Methyltriphenylmethane.

### 1 : 1 : 2-Triphenylethane



C<sub>20</sub>H<sub>18</sub>

MW, 258

Leaflets from EtOH. M.p. 54.5°. B.p. 348-9°, 216-17°/14 mm. Forms a K salt.

Böeseken, Bastet, *Rec. trav. chim.*, 1913, 32, 199.

Klages, Heilmann, *Ber.*, 1904, 37, 1455.

### Triphenylethyl Alcohol.

See Hydroxytriphenylethane.

**1 : 1 : 2-Triphenylethylene** ( $\alpha$ -Phenylstilbene)

$C_{20}H_{16}$   $C_6H_5 \cdot CH : C(C_6H_5)_2$  MW, 256

Leaflets from EtOH or AcOH. M.p. 72-3°. B.p. 220-1°/14 mm. Heat of comb.  $\bullet C_v$  2510.3 Cal.

Stadnikow, *Ber.*, 1914, 47, 2140.

Staudinger, *Kon. Ann.*, 1911, 384, 89.

**2 : 4 : 5-Triphenylglyoxaline.**

See Lophine.

 **$\alpha$ -Triphenylguanidine**

$C_{19}H_{17}N_3$   $C_6H_5 \cdot NH \cdot \overset{N \cdot C_6H_5}{\underset{|}{C}} \cdot NH \cdot C_6H_5$  MW, 287

Prisms from EtOH. M.p. 144-144.5°.  $D^{19}_D$  1.163. Dist. in  $CO_2 \rightarrow$  carbanilide. Conc. KOH  $\rightarrow CO_2$  + aniline. Rubber vulcanisation accelerator (T.P.G.).

$B, HCl$ : prisms +  $1H_2O$ . M.p. 241-2°.

*Picrate*: yellow cryst. from EtOH. M.p. 180°.

Connolly, Dyson, *J. Chem. Soc.*, 1935, 680.

Hofmann, *Ber.*, 1869, 2, 458.

Alway, Vail, *Am. Chem. J.*, 1902, 28, 162.

 **$\beta$ -Triphenylguanidine**

$C_{19}H_{17}N_3$   $C_6H_5 \cdot NH \cdot \overset{NH}{\underset{|}{C}} \cdot N(C_6H_5)_2$  MW, 287

Plates. M.p. 131°. Sol. EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ .

v. Braun, *Ber.*, 1900, 33, 2725.

Weith, Schröder, *Ber.*, 1875, 8, 295.

**Triphenylhexahydrotriazine.**

See Anhydroformaldehydeaniline.

**Triphenylmethane (Tritan)**

$(C_6H_5)_2CH \cdot C_6H_5$  MW, 244

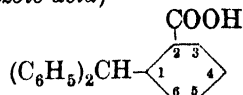
Cryst. from EtOH in two forms. (i) Labile, m.p. 81°. (ii) Stable, m.p. 94°. B.p. 190-215°/10 mm.  $D^{20}_D$  1.01405. Sol.  $Et_2O$ ,  $CHCl_3$ , hot EtOH. Spar. sol. AcOH. Very spar. sol. ligroin. Heat of comb.  $C_p$  2387.3 Cal.,  $C_v$  2385.1 Cal.  $CrO_3 \rightarrow$  triphenylcarbinol + benzophenone.  $PCl_5 \rightarrow$  triphenylchloromethane.

Norris, *Organic Syntheses*, Collective Vol. I, 532.

Schmidlin, Garcia-Banús, *Ber.*, 1912, 45, 3189.

**Triphenylmethane- $\alpha$ -carboxylic Acid.**

See Triphenylacetic Acid.

**Triphenylmethane-2-carboxylic Acid (2-Benzhydrylbenzoic acid)**

$C_{20}H_{16}O_2$  MW, 288

Needles from EtOH. M.p. 162°. Sublimes. Sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ . Spar. sol. ligroin. Insol.  $H_2O$ .

*Me ester*:  $C_{21}H_{18}O_2$ . MW, 302. Prisms from MeOH. M.p. 98°.

*Nitrile*: 2-cyanotriphenylmethane.  $C_{20}H_{15}N$ . MW, 269. Needles from EtOH. M.p. 89°. B.p. 270-85°/20-30 mm. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ . Insol. ligroin.

Drory, *Ber.*, 1891, 24, 2573.

Gresly, *Ann.*, 1886, 234, 242.

**Triphenylmethane-4-carboxylic Acid (4-Benzhydrylbenzoic acid).**

Needles from AcOH.Aq. M.p. 165°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Prac. insol.  $H_2O$ .

*Chloride*:  $C_{20}H_{15}OCl$ . MW, 306.5. Cryst. from pet. ether. M.p. 89-90°.

*Nitrile*: 4-cyanotriphenylmethane. Prisms from MeOH. M.p. 100°. Sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ .

*Anilide*: cryst. from AcOH. M.p. 196°.

Staudinger, Clar, *Ber.*, 1911, 44, 1628.

Moses, *Ber.*, 1900, 33, 2630.

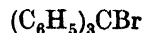
**Triphenylmethyl.**

See Hexaphenylethane.

 **$\omega$ -Triphenylmethyl-benzyl Alcohol.**

See 1 : 2 : 2 : 2-Tetraphenylethyl Alcohol.

**Triphenylmethyl bromide** ( $\alpha$ -Bromotriphenylmethane, trityl bromide, triphenylbromomethane)



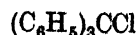
$C_{19}H_{15}Br$  MW, 323

Pale yellow cryst. from  $CS_2$ . M.p. 152°. B.p. 230°/15 mm. Boiling AcOH  $\rightarrow$  triphenylcarbinol.  $NH_3$  in  $C_6H_6$  sol.  $\rightarrow$  triphenylmethylaniline.

Allen, Kölliker, *Ann.*, 1885, 227, 110.

Wieland, *Ber.*, 1909, 42, 3024 (Footnote).

**Triphenylmethyl chloride** ( $\alpha$ -Chlorotriphenylmethane, triphenylchloromethane, trityl chloride)



$C_{19}H_{15}Cl$  MW, 278.5

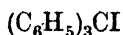
Cryst. from  $C_6H_6$  or pet. ether. M.p. 112-13°. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Heat of

comb.  $C_p$  2346.5 Cal.,  $C_p$  2348.5 Cal. Above  $250^\circ \rightarrow$  triphenylmethane + a little 9-phenylfluorene. Employed for characterisation of alcohols as triphenylmethyl ethers.

Gomberg, *Ber.*, 1900, **33**, 3147.

Norris, Sanders, *Am. Chem. J.*, 1901, **25**, 60.

**Triphenylmethyl iodide** ( $\alpha$ -Iodotriphenylmethane, trityl iodide)



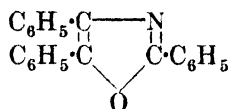
$C_{19}H_{15}I$  MW, 370

Yellowish cryst. which rapidly turn dark brown. M.p.  $132^\circ$ .

$C_{19}H_{15}I, 5I$ : prisms. M.p.  $90^\circ$ .

Gomberg, *Ber.*, 1900, **33**, 3158; 1902, **35**, 1835.

**2 : 4 : 5-Triphenyloxazole** (*Benzilam, azobenzil*)



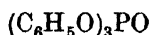
$C_{21}H_{15}ON$  MW, 297

Prisms. M.p.  $115.5\text{--}116.5^\circ$ . Sol.  $C_6H_6$  with feeble blue fluor.  $CrO_3 \rightarrow$  benzoic acid.

Schönberg, *Ber.*, 1921, **54**, 242.

McKenzie, Barrow, *J. Chem. Soc.*, 1913, **103**, 1334.

**Triphenyl phosphate**



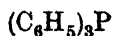
$C_{18}H_{15}O_4P$  MW, 326

Prisms from EtOH. M.p.  $49^\circ$ . B.p.  $245^\circ/11$  mm. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. EtOH. Insol.  $H_2O$ .

Heim, *Ber.*, 1883, **16**, 1765.

A.G.F.A., D.R.P., 246,871, (*Chem. Zentr.*, 1912, I, 1875).

**Triphenylphosphine**



$C_{18}H_{15}P$  MW, 262

Plates or prisms from  $Et_2O$ . M.p.  $80^\circ$ . Dist. undecomp. above  $360^\circ$  in indifferent atmosphere.  $n_D^{20}$  1.52475. Very sol.  $Et_2O$ . Sol.  $CHCl_3$ , AcOH,  $C_6H_6$ . Mod. sol. EtOH. Insol.  $H_2O$ . Heat of comb.  $C_p$  2480.7 Cal.,  $C$ , 2483.95 Cal. Triboluminescent.

$B, HI$ : needles. M.p.  $215^\circ$  decomp.

*Methiodide*: leaflets. M.p.  $182\text{--}3^\circ$ .

*Ethiodide*: leaflets from EtOH.Aq. M.p.  $164\text{--}5^\circ$ .

$B, C_6H_5Br$ : tetraphenylphosphonium bromide. M.p.  $286\text{--}8^\circ$ .

$B, C_6H_5Cl$ : tetraphenylphosphonium chloride. M.p.  $265^\circ$ .

$B, C_6H_5I$ : tetraphenylphosphonium iodide. M.p.  $333^\circ$ .

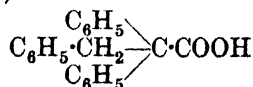
Dodonow, Medox, *Ber.*, 1928, **61**, 907.

Michaelis, v. Soden, *Ann.*, 1885, **229**, 298.

**Triphenyl phosphite.**

See under Phenol.

**1 : 1 : 2-Triphenylpropionic Acid** (*Diphenylbenzylacetic acid*, 1 : 1-diphenylhydrocinnamic acid)



$C_{21}H_{18}O_2$  MW, 302

Needles from EtOH.Aq. M.p.  $162^\circ$  ( $132^\circ$ ). Sol.  $Et_2O$ , hot EtOH.Aq. Very spar. sol.  $H_2O$ .

*Me ester*:  $C_{22}H_{20}O_2$ . MW, 316. M.p.  $127^\circ$ .

*Benzyl ester*: prisms from EtOH. M.p.  $85^\circ$ . B.p.  $270^\circ/2$  mm.

*Chloride*:  $C_{21}H_{17}OCl$ . MW, 320.5. M.p.  $90\text{--}1^\circ$ .

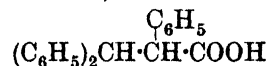
*Amide*:  $C_{21}H_{19}ON$ . MW, 301. Needles from EtOH. M.p.  $111^\circ$ .

*Nitrile*:  $C_{21}H_{17}N$ . MW, 283. Plates or needles. M.p.  $126^\circ$ .

Ramart, *Bull. soc. chim.*, 1924, **35**, 196.

Neure, *Ann.*, 1889, **250**, 143, 147.

**1 : 2 : 2-Triphenylpropionic Acid** (*Phenylbenzhydrylacetic acid*)



$C_{21}H_{18}O_2$  MW, 302

Needles from EtOH or pet. ether. M.p.  $222\text{--}3^\circ$  ( $211^\circ$ ). Sol. EtOH,  $Et_2O$ . Spar. sol. ligroin. Insol.  $H_2O$ .

*Me ester*: plates from EtOH. M.p.  $159^\circ$ .

*Et ester*:  $C_{23}H_{22}O_2$ . MW, 330. Leaflets from EtOH. M.p.  $122\text{--}3^\circ$ .

*Propyl ester*:  $C_{24}H_{24}O_2$ . MW, 344. M.p.  $109^\circ$ .

*Isopropyl ester*: m.p.  $138^\circ$ .

*Phenyl ester*:  $C_{27}H_{22}O_2$ . MW, 378. M.p.  $124^\circ$ .

*Benzyl ester*: m.p.  $117^\circ$ .

*Chloride*: cryst. from pet. ether. M.p.  $94^\circ$ .

*Amide*: needles from EtOH.Aq. M.p.  $213^\circ$ .



**Nitrile** : plates from MeOH. M.p. 102°.

Kohler, Heritage, *Am. Chem. J.*, 1905, **33**, 156.

Banús, Salas, *Chem. Zentr.*, 1935, II, 3770.

Banús, Boqué, *ibid.*

**2 : 2 : 2-Triphenylpropionic Acid** (2 : 2-Diphenylhydrocinnamic acid)

$(C_6H_5)_3C \cdot CH_2 \cdot COOH$   
 $C_{21}H_{18}O_2$  MW, 302

Prisms from EtOH. M.p. 177°. Very sol.  $Et_2O$ . Sol. EtOH.

*Et ester* : prisms. M.p. 125° (81°).

*Chloride* : m.p. 128-9° (132°).

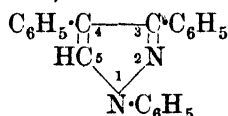
*Amide* : m.p. 198°.

*Nitrile* : cryst. from EtOH. M.p. 140°.

Fosse, *Bull. soc. chim.*, 1931, **49**, 159.

Henderson, *J. Chem. Soc.*, 1887, **51**, 226.

**1 : 3 : 4-Triphenylpyrazole** (1 : 3 : 4-Triphenyl-1 : 2-diazole)



$C_{21}H_{16}N_2$  MW, 296

Needles from EtOH. M.p. 207° (185°). Very sol.  $C_6H_6$ . Spar. sol. EtOH, ligroin, cold AcOH. Insol. dil. acids. Sol. conc.  $H_2SO_4$  with yellow col.

Rupe, Gisiger, *Helv. Chim. Acta*, 1925, **8**, 351.

**1 : 3 : 5-Triphenylpyrazole.**

Needles or plates from  $Et_2O$ . M.p. 140-140.5° (137-8°). Sol.  $CHCl_3$ , AcOH,  $C_6H_6$ . Spar. sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

*Methiodide* : needles from  $H_2O$ . M.p. 176° → components.

Knorr, Laubmann, *Ber.*, 1888, **21**, 1206.

Wislicenus, *Ann.*, 1899, **308**, 253.

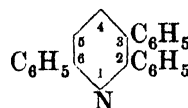
**1 : 4 : 5-Triphenylpyrazole.**

Needles from EtOH or AcOH. M.p. 212° (206°). B.p. about 400° undecomp. Sol.  $CHCl_3$ , hot AcOH, hot  $C_6H_6$ . Spar. sol.  $Et_2O$ , hot EtOH.

Japp, Tingle, *J. Chem. Soc.*, 1897, **71**, 1143.

Wislicenus, Ruthing, *Ann.*, 1911, **379**, 257.

**2 : 3 : 6-Triphenylpyridine**



$C_{23}H_{17}N$

MW, 307

Needles from EtOH. M.p. 115°.

*Picrate* : yellow prisms from EtOH. M.p. 163°.

Allen, Barker, *J. Am. Chem. Soc.*, 1932, **54**, 742.

**2 : 4 : 6-Triphenylpyridine (Acetophenine).**

Colourless prisms from EtOH. M.p. 137.5° (135°). Very spar. sol. cold EtOH. Sol. conc.  $H_2SO_4$  with blue fluor.

*Picrate* : yellow needles from EtOH. M.p. 192.5°.

Reddélien, *Ber.*, 1920, **53**, 334.

**Triphenylstibine (Antimony triphenyl)**

$(C_6H_5)_3Sb$

$C_{18}H_{15}Sb$

MW, 351

Prisms from pet. ether. M.p. 53° (50°). B.p. above 220°/1 mm.  $D_4^{50}$  1.4075,  $D_4^{100}$  1.3597. Very sol.  $Et_2O$ ,  $CHCl_3$ , AcOH,  $C_6H_6$ , pet. ether. Spar. sol. EtOH.

Hiers, *Organic Syntheses*, Collective Vol. I, 535.

**Triphenylthiourea.**

*See under* Thiourea.

**Triphenylurea.**

*See under* Urea.

**Tripopin.**

*See under* Propionic Acid.

**Tripropylamine**

$(CH_3 \cdot CH_2 \cdot CH_2)_3N$

$C_9H_{21}N$

MW, 143

Liq. B.p. 156.5°.  $D_4^{20}$  0.753.  $n_D^{19.4}$  1.41756.  $k = 4.43 (5.5) \times 10^{-4}$  at 25°.

*B, HCl* : hygroscopic needles. M.p. 90°.

*B, HBr* : needles. M.p. 180°.

*B, HAuBr\_4* : m.p. 149°.

*B, HBr, HgBr\_2* : needles. M.p. 104°.

*Methiodide* : methyltripropylammonium iodide. Leaflets from EtOH- $Et_2O$ . M.p. 207-8°.

*Ethiodide* : ethyltripropylammonium iodide. Prisms from EtOH. M.p. 238° decomp.

*B, I\_2* : cryst. from EtOH. M.p. 65-6°.

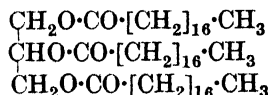
Skita, Keil, *Monatsh.*, 1929, **53** and **54**, 758.

**Tripropyl phosphate.**

See under Phosphoric Acid.

**Trisalicilin.**

See under Salicylic Acid.

**Tristearin** (*Glycerol tristearate, stearin*) $\text{C}_{57}\text{H}_{110}\text{O}_6$ 

MW, 890

Present in many natural fats. Cryst. from  $\text{Et}_2\text{O}$ . M.p.  $72^\circ$  ( $70\text{--}8^\circ$ ).  $D_4^{20}$  0.8559.  $n_D^{20}$  1.4385. Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , hot  $\text{EtOH}$ . Mod. sol. boiling  $\text{Et}_2\text{O}$ , boiling pet. ether. Insol. cold  $\text{EtOH}$ .

Bellucci, *Gazz. chim. ital.*, 1912, 42, ii, 291.

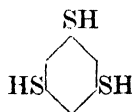
**Tritan.**

See Triphenylmethane.

**Tritanol.**

See Triphenylcarbinol.

**Trithiophloroglucinol** (1 : 3 : 5-*Trimercaptobenzene*)

 $\text{C}_6\text{H}_6\text{S}_3$ 

MW, 174

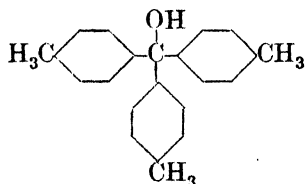
Needles. M.p.  $57\text{--}60^\circ$ . Sol.  $\text{Et}_2\text{O}$ ,  $\text{CS}_2$ .

*Tri-Me ether*:  $\text{C}_9\text{H}_{12}\text{S}_3$ . MW, 216. Needles from  $\text{EtOH}$ . M.p.  $66\text{--}8^\circ$ .

*Triacetyl*: needles from  $\text{EtOH}$ . M.p.  $73\text{--}4^\circ$ .

Pollak, *Carniol, Ber.*, 1909, 42, 3252.

**Tri-*p*-tolylcarbinol** ( $\alpha$ -*Hydroxy-4 : 4' : 4''-trimethyltriphenylmethane*)

 $\text{C}_{22}\text{H}_{22}\text{O}$ 

MW, 302

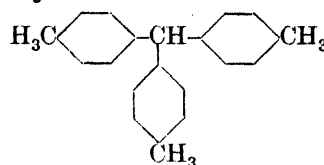
Prisms from  $\text{Et}_2\text{O}$ -ligroin. M.p.  $96^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. pet. ether.  $\text{H}_2\text{SO}_4 \rightarrow$  greenish-red col.

*Et ether*:  $\text{C}_{24}\text{H}_{26}\text{O}$ . MW, 330. Needles from pet. ether. M.p.  $111^\circ$ .

Gomberg, *Ber.*, 1904, 37, 1629.

Mothwurf, *ibid.*, 3153.

Tousley, Gomberg, *J. Am. Chem. Soc.*, 1904, 26, 1516.

**Tri-*p*-tolylmethane** $\text{C}_{22}\text{H}_{22}$ 

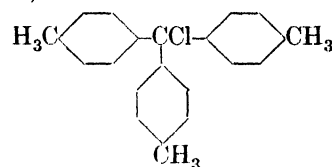
MW, 286

Prisms. M.p.  $63^\circ$ . B.p.  $225\text{--}30^\circ/15$  mm. Spar. sol.  $\text{EtOH}$ ,  $\text{AcOH}$ .

Tousley, Gomberg, *J. Am. Chem. Soc.*, 1904, 26, 1520.

Mothwurf, *Ber.*, 1904, 37, 3155.

**Tri-*p*-tolylmethyl chloride** ( $\alpha$ -*Chlorotri-p-tolylmethane*)

 $\text{C}_{22}\text{H}_{21}\text{Cl}$ 

MW, 320.5

Cryst. from  $\text{AcOEt}$ . M.p.  $184\text{--}5^\circ$  ( $173^\circ$ ). Sol.  $\text{C}_6\text{H}_6$ ,  $\text{CS}_2$ , pet. ether.  $\text{H}_2\text{SO}_4 \rightarrow$  tri-*p*-tolylcarbinol.

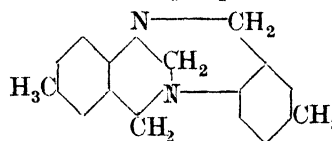
Mothwurf, *Ber.*, 1904, 37, 3156.

Gomberg, *ibid.*, 1627.

**Tritopine.**

See Laudanidine.

**Tröger's Base** (1 : 2'-*Methylene-3-p-tolyl-6-methyl-1 : 2 : 3 : 4-tetrahydroquinazoline*)

 $\text{C}_{17}\text{H}_{18}\text{N}_2$ 

MW, 250

Needles from  $\text{EtOH}$ . Aq. M.p.  $135\text{--}6^\circ$ .

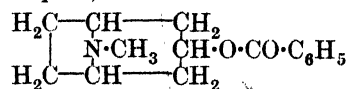
*Hydrochloride*: m.p.  $213^\circ$ .

*Picrate*: cryst. from  $\text{EtOH}$ . M.p.  $188\text{--}9^\circ$ .

Wagner, *J. Am. Chem. Soc.*, 1935, 57, 1296.

Spielman, *ibid.*, 583.

**Tropacocaine** ( $\psi$ -*Tropyl ester of benzoic acid, benzoyl- $\psi$ -tropein*)

 $\text{C}_{15}\text{H}_{19}\text{O}_2\text{N}$ 

MW, 245

Constituent of Java coca leaves. Plates. M.p.  $49^\circ$ . Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , ligroin,

$C_6H_6$ . Hot conc.  $HCl \rightarrow \psi$ -tropine + benzoic acid.  $H_2O_2 \rightarrow N$ -oxide. Resorcinol + conc.  $H_2SO_4 \rightarrow$  yellow  $\rightarrow$  violet  $\rightarrow$  red col. 2-Naphthol + conc.  $H_2SO_4 \rightarrow$  grey  $\rightarrow$  dark blue col. Produces paralysis of central nervous system. Poisonous. Exhibits local anæsthetic properties.

$B, HCl$ : plates from EtOH. M.p.  $283^\circ$ .

$B, HAuCl_4$ : yellow needles from  $H_2O$ . M.p.  $208^\circ$ .

$B_2, H_2PtCl_6$ : yellow needles. M.p.  $276-8^\circ$ .

$d$ -Camphor- $\beta$ -sulphonic acid salt: prisms from EtOH-AcOEt. M.p.  $176-7^\circ$ .  $[\alpha]_D + 11.1^\circ$  in  $H_2O$ .

$\alpha$ -Bromo-[ $d$ -camphor]- $\pi$ -sulphonic acid salt: prisms from EtOH, m.p.  $190^\circ$ ; needles +  $3H_2O$  from  $H_2O$ , m.p.  $73^\circ$ .  $[\alpha]_D + 47.3^\circ$  in  $H_2O$ .

$B, C_6H_5 \cdot COOH$ : m.p.  $60-1^\circ$ .

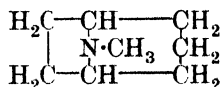
$N$ -Oxide:  $C_{15}H_{19}O_3N$ . MW, 261. Cryst. M.p.  $152-3^\circ$ . Hydrochloride: m.p.  $200^\circ$ .

Picrate: needles. Darkens at  $215-20^\circ$ , m.p.  $240-2^\circ$ .

Willstätter, *Ber.*, 1896, **29**, 943.

Hesse, *Ann.*, 1892, **271**, 208.

### Tropane (Dihydrotropidine)



$C_8H_{15}N$

MW, 125

Liq. B.p.  $167^\circ$ .  $D_4^{20}$  0.931. Misc. with cold  $H_2O$ . Spar. misc. with hot  $H_2O$ .  $H_2O_2 \rightarrow$  tropane- $N$ -oxide. Dist. of hydrochloride in current of  $HCl \rightarrow$  nortropine.

$B, HAuCl_4$ : plates from EtOH. M.p.  $245-6^\circ$ .

$B_2, H_2PtCl_6$ : orange-yellow plates from  $H_2O$ . Decomp. at  $229-30^\circ$ .

$N$ -Oxide:  $C_8H_{15}ON$ . MW, 141. Hygroscopic cryst.  $B_2, H_2PtCl_6$ : orange-yellow needles. M.p.  $228^\circ$  decomp.

Methiodide: cryst. from  $H_2O$ . Does not melt below  $300^\circ$ .

Picrate: needles. M.p.  $281^\circ$  decomp.

Hess, *Ber.*, 1918, **51**, 1007.

### Tropanol.

See Tropine.

### $\psi$ -Tropanol.

See  $\psi$ -Tropine.

### Tropanone.

See Tropinone.

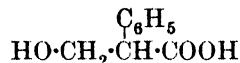
### Tropeïn.

See under Tropine.

### Tropene.

See Tropidine.

**Tropic Acid** (1-Phenylhydracrylic acid,  $\beta$ -hydroxyhydratropic acid, 2-hydroxy-1-phenylpropionic acid,  $\alpha$ -hydroxymethylphenylacetic acid)



$C_9H_{10}O_3$

MW, 166

$d$ .

Needles, scales or prisms from  $H_2O$ , needles from  $C_6H_6$ . M.p.  $129-30^\circ$ .  $[\alpha]_D^{15} + 72.2^\circ$  in EtOH,  $[\alpha]_D + 81.6^\circ$  in  $H_2O$ .

Quinine salt: needles or plates from EtOH. M.p.  $191.5-192.5^\circ$ . Spar. sol.  $H_2O$ .  $[\alpha]_D - 104^\circ$  in EtOH.

$l$ .

Needles from  $H_2O$ , plates from AcOEt. M.p.  $129-30^\circ$ . Spar. sol. cold  $C_6H_6$ . Sol. AcOEt, methyl ethyl ketone.  $[\alpha]_D^{15} - 72.5^\circ$  in EtOH,  $[\alpha]_D - 81.2^\circ$  in  $H_2O$ .

Quinine salt: plates or needles from EtOH. M.p.  $185-6^\circ$ . More sol. EtOH than  $d$ -salt.  $[\alpha]_D - 140.7^\circ$  in EtOH.

Tropine ester: see Hyoscyamine.

Scopine ester: see Hyoscine.

$dl$ .

Needles or plates from  $H_2O$ , cryst. from  $C_6H_6$ . M.p.  $118^\circ$ . Sol. 49 parts  $H_2O$  at  $14.5^\circ$ . Very sol. hot  $H_2O$ . Sol. EtOH, Et<sub>2</sub>O. Spar. sol.  $C_6H_6$ . Insol.  $CS_2$ .  $k = 7.50 \times 10^{-5}$  at  $25^\circ$ . Resolved into active components through quinine salt. Heat at  $160^\circ \rightarrow$  tropide. Ox.  $\rightarrow C_6H_5 \cdot CHO + C_6H_5 \cdot COOH$ . Boiling aq.  $Ba(OH)_2$  or aq.  $KOH \rightarrow$  atropic acid. Conc.  $HCl$  at  $140^\circ \rightarrow \alpha$ -isatropic acid.

Me ester:  $C_{10}H_{12}O_3$ . MW, 180. Needles. M.p.  $36.5-37.5^\circ$ . B.p.  $159-62^\circ/19$  mm.

Tropine ester: see Atropine.

Amide:  $C_9H_{11}O_2N$ . MW, 165. Plates and needles from  $H_2O$ . M.p.  $169.5^\circ$ . Sol. hot  $H_2O$ . Spar. sol. cold  $H_2O$ .

Me ether:  $C_{10}H_{12}O_3$ . MW, 180. Cryst. from petrol. M.p.  $62^\circ$ . B.p.  $134-6^\circ/0.3$  mm.

Acetyl: plates. M.p.  $88-90^\circ$ .

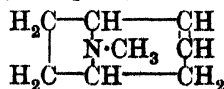
Chambon, *Compt. rend.*, 1928, **186**, 1630.

King, Palmer, *J. Chem. Soc.*, 1922, **121**, 2577.

McKenzie, Wood, *J. Chem. Soc.*, 1919, **115**, 838.

Wislicenus, Bilhuber, *Ber.*, 1918, **51**, 1237.

### Tropidine (2-Tropene)



$C_8H_{13}N$

MW, 123

Liq. B.p. 163°. Sol. EtOH. Et<sub>2</sub>O. Spar. misc. with hot H<sub>2</sub>O. Mod. misc. with cold H<sub>2</sub>O.  $D_4^{20}$  0.953.  $n_D^{19}$  1.4884. Volatile in steam. H<sub>2</sub>O<sub>2</sub>  $\rightarrow$  *N*-oxide. Red.  $\rightarrow$  tropane. Stable to hot H<sub>2</sub>SO<sub>4</sub> and alkalis.

*B,HAuCl<sub>4</sub>*: m.p. 212°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-red cryst. Decomp. at 217°.

*N-Oxide*: C<sub>8</sub>H<sub>13</sub>ON. MW, 139. Hygroscopic cryst. *B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-yellow plates from H<sub>2</sub>O. M.p. about 220° decomp.

*Methiodide*: cryst. from H<sub>2</sub>O or EtOH. M.p. about 300°.

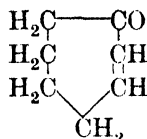
*Picrate*: yellow needles from H<sub>2</sub>O. M.p. about 285° decomp.

Willstätter, *Ber.*, 1901, 34, 142.

### Tropigenin.

See Nortropine.

### Tropilene (Cycloheptenone-3)



C<sub>7</sub>H<sub>10</sub>O

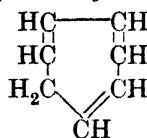
MW, 110

Liq. B.p. 186–8°. Insol. H<sub>2</sub>O.  $D_4^{20}$  1.0091. Reduces NH<sub>3</sub>.AgNO<sub>3</sub> and Fehling's. Ox.  $\rightarrow$  adipic and oxalic acids. Zn + AcOH  $\rightarrow$  methylcyclohexanone. Catalytic red.  $\rightarrow$  suberone.

*Oxime*: cryst. from ligroin. M.p. 80–8°.

Kötz, Blendermann, Mähner, Rosenbusch, *Ann.*, 1913, 400, 80.

### Tropilidene ( $\Delta^{1:3:5}$ -Cycloheptatriene)



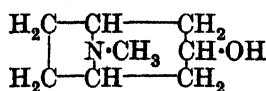
C<sub>7</sub>H<sub>8</sub>

MW, 92

Liq. B.p. 117°/749 mm.  $D_4^{18.5}$  0.8875.  $n_D^{17.5}$  1.51751. Resinifies on standing in air. Alc. sol. + conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  dark brownish-red col.

Willstätter, *Ann.*, 1901, 317, 204, 269.

### Tropine (3-Tropanol)



C<sub>8</sub>H<sub>15</sub>ON

MW, 141

Hygroscopic plates from Et<sub>2</sub>O. M.p. 63°. B.p. 229°. Very sol. H<sub>2</sub>O, EtOH. Difficultly

volatile in steam.  $k = 2.74 \times 10^{-4}$  at 25°. NaOEt  $\rightarrow$   $\psi$ -tropine. H<sub>2</sub>O<sub>2</sub>  $\rightarrow$  *N*-oxide. CrO<sub>3</sub>  $\rightarrow$  tropinone. KMnO<sub>4</sub> in alk. sol.  $\rightarrow$  nortropine. Acid KMnO<sub>4</sub>  $\rightarrow$  tropinone. H<sub>2</sub>SO<sub>4</sub> + AcOH  $\rightarrow$  tropidine. Soda-lime dist.  $\rightarrow$  tropilidene. HI + PH<sub>4</sub>I  $\rightarrow$  tropane.

*B,HAuCl<sub>4</sub>*: yellow plates. M.p. 210–12° decomp.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellowish-red plates from H<sub>2</sub>O. M.p. 197–8° decomp.

*d-Camphor- $\beta$ -sulphonic acid salt*: plates from EtOH-AcOEt or CHCl<sub>3</sub>. M.p. 236°.  $[\alpha]_D + 32.1^\circ$  in CHCl<sub>3</sub>.

*Picrate*: yellow needles from H<sub>2</sub>O. Decomp. at about 275°.

*Me ether*: C<sub>9</sub>H<sub>17</sub>ON. MW, 155. Cryst. powder. Does not melt below 300°. Very sol. H<sub>2</sub>O, hot EtOH. Spar. sol. Et<sub>2</sub>O.

*Acetyl*: acetyltropine. Liq. B.p. 235–7°.

*Benzoyl*: benzoyltropine. Plates + 2H<sub>2</sub>O, m.p. 56°: anhyd. cryst. from Et<sub>2</sub>O, m.p. 41–2°. B.p. 175–80°. *B,HCl*: prisms from EtOH. M.p. 275°.

*N-Oxide*: C<sub>9</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 157. M.p. 238°.

*Mandelic ester*: see Homatropine.

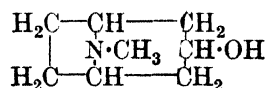
*l-Tropic ester*: see Hyoscyamine.

*dl-Tropic ester*: see Atropine.

*Phenylcarbamate ester*: see Uretropine.

Willstätter, *Ber.*, 1902, 35, 1870.

### $\psi$ -Tropine ( $\psi$ -3-Tropanol)



C<sub>8</sub>H<sub>15</sub>ON

MW, 141

Stereoisomer of tropine. Prisms from C<sub>6</sub>H<sub>6</sub>-pet. ether or ligroin. M.p. 108–9°. B.p. 240–1°. Very sol. H<sub>2</sub>O, EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Sol. Et<sub>2</sub>O. Ox.  $\rightarrow$  tropinone. Dil. alk. KMnO<sub>4</sub>  $\rightarrow$  nor- $\psi$ -tropine. H<sub>2</sub>O<sub>2</sub>  $\rightarrow$  *N*-oxide. AcOH + H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  tropidine.

*B,HCl*: needles from EtOH. Sinters at 250°. M.p. 280–2° decomp.

*B,HAuCl<sub>4</sub>*: yellow plates or needles from H<sub>2</sub>O. M.p. 225° decomp.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: orange-red plates + 4H<sub>2</sub>O from H<sub>2</sub>O. M.p. anhyd. 206°.

*d-Camphor- $\beta$ -sulphonic acid salt*: prisms from EtOH-AcOEt. M.p. 224–6°.  $[\alpha]_D + 26.3^\circ$  in EtOH.

*Picrate*: needles or prisms from H<sub>2</sub>O. Darkens at 245°, m.p. 258–9° decomp.

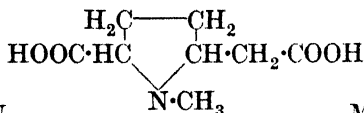
*Benzoyl*: see Tropacocaine.

*N-Oxide*: C<sub>9</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 157. M.p. 229°.

**Hydrochloride**: m.p. 286°. **Picrate**: m.p. 257° decomp.

Willstätter, *Ber.*, 1901, **34**, 3165.

**Tropinic Acid** (*N*-Methylpyrrolidine-5-carboxylic acid-2-acetic acid)



$\text{C}_8\text{H}_{13}\text{O}_4\text{N}$  MW, 187  
d.

Cryst. from  $\text{H}_2\text{O}$  or EtOH.Aq. M.p. (rapid heat.) 253° decomp., (slow heat.) 247–8°. Sol. cold  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .  $[\alpha]_D^{20} + 14.8^\circ$  in  $\text{H}_2\text{O}$ . Decolourises  $\text{KMnO}_4$  sol. in cold.

**Di-Me ester**:  $\text{C}_{10}\text{H}_{17}\text{O}_4\text{N}$ . MW, 215. Oil. **Picrate**: needles. M.p. 120–1°. **Methiodide**: plates and needles from MeOH. M.p. 176–7° decomp.

l.

Cryst. from  $\text{H}_2\text{O}$ . M.p. 243°.  $[\alpha]_D^{20} - 14.8^\circ$  in  $\text{H}_2\text{O}$ .

dl.

Needles from EtOH.Aq. M.p. about 248° decomp. Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Resolved through cinchonine salt. Loses  $\text{CO}_2$  on heating above m.p. Decolourises  $\text{KMnO}_4$  sol. in cold.  $\text{CrO}_3 + \text{H}_2\text{SO}_4 \rightarrow \text{N}$ -methylsuccinimide.  $\text{HI}(+ \text{P}) \rightarrow \text{N}$ -methylpyrrolidine.

$\text{B}_2\text{H}_2\text{PtCl}_6$ : orange-yellow cryst. Decomp. at 100–10°.

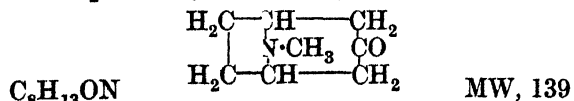
**Di-Me ester**:  $\text{C}_{10}\text{H}_{17}\text{O}_4\text{N}$ . MW, 215. Oil. B.p. 268–72° (part. decomp.). **Picrate**: orange-yellow prisms from EtOH. M.p. 121°. **Methiodide**: prisms +  $\frac{1}{2}\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$  or EtOH.Aq., anhyd. plates from EtOH– $\text{Et}_2\text{O}$ . M.p. 171–2° decomp.

**Di-Et ester**:  $\text{C}_{11}\text{H}_{21}\text{O}_4\text{N}$ . MW, 243. Oil. B.p. 160°/18.5 mm.

Willstätter, *Ber.*, 1898, **31**, 1547.

Liebermann, *Ber.*, 1891, **24**, 2587.

### Tropinone (3-Trovanone)



$\text{C}_8\text{H}_{13}\text{ON}$  MW, 139

Needles from pet. ether. M.p. 42°. B.p. 224–5°, 125°/40 mm., 113°/25 mm. Very sol. most org. solvents. Reacts alkaline. Reduces warm  $\text{AgNO}_3$ .  $\text{H}_2\text{O} \rightarrow \text{N}$ -oxide.

$\text{B}, \text{HCl}$ : prisms from EtOH. M.p. about 188–9° decomp.

$\text{B}, \text{HAuCl}_4$ : yellow prisms from dil. HCl. M.p. about 163° decomp.

$\text{B}_2\text{H}_2\text{PtCl}_6$ : orange-red prisms. M.p. 191–2° decomp.

d-Camphor- $\beta$ -sulphonic acid salt: cryst. from AcOEt. M.p. 216° decomp.

**Picrate**: yellow needles from  $\text{H}_2\text{O}$ . M.p. 220° decomp.

**N-Oxide**:  $\text{C}_8\text{H}_{13}\text{O}_2\text{N}$ . MW, 155. M.p. 98°.

**Oxime**: prisms from ligroin. M.p. 115–16°. Very sol.  $\text{H}_2\text{O}$ , EtOH, warm  $\text{Me}_2\text{CO}$ , AcOEt,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ .  $\text{B}, \text{HCl}$ : prisms from EtOH. M.p. 242° decomp. **Methiodide**: prisms from EtOH.Aq. M.p. 236° decomp.

**Semicarbazone**: plates from EtOH. M.p. 212–13°.

**Methiodide**: cryst. from  $\text{H}_2\text{O}$ . M.p. 273–5°.

**Dibenzylidene deriv.**: yellow prisms. M.p. 152°.

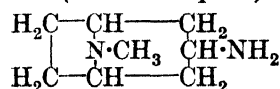
Schöpf, Lehmann, *Ann.*, 1935, **518**, 1.

Willstätter, Bommer, *Ann.*, 1926, **422**, 30.

Robinson, *J. Chem. Soc.*, 1917, **111**, 766.

Willstätter, *Ber.*, 1896, **29**, 396, 947; 1898, **31**, 1540; 1900, **33**, 1169.

### Tropylamine (3-Aminotropane)



$\text{C}_8\text{H}_{16}\text{N}_2$  MW, 140

Liq. B.p. 211°, 91–2°/12 mm. Hot Na amylate  $\rightarrow \psi$ -tropylamine.

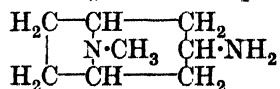
$\text{B}_2\text{H}_2\text{AuCl}_4$ : prisms and plates. M.p. 220–1° decomp.

$\text{B}_2\text{H}_2\text{PtCl}_6$ : red plates from  $\text{H}_2\text{O}$ . M.p. 257° decomp.

**Picrate**: plates. M.p. 235° decomp.

Willstätter, Müller, *Ber.*, 1898, **31**, 1211.

### $\psi$ -Tropylamine ( $\psi$ -3-Aminotropane)



$\text{C}_8\text{H}_{16}\text{N}_2$  MW, 140

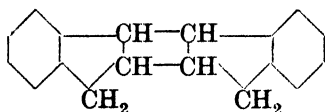
Stereoisomer of tropylamine. Liq. B.p. 213°, 107°/26 mm., 98–100°/17.5 mm. Sol.  $\text{H}_2\text{O}$  with heat evolution. Sol. EtOH,  $\text{Et}_2\text{O}$ . Strong base. Absorbs  $\text{CO}_2$  from the air. Stable to hot Na amylate.

$\text{B}_2\text{H}_2\text{AuCl}_4$ : plates, needles or prisms, with or without  $\text{H}_2\text{O}$  from  $\text{H}_2\text{O}$ . M.p. 223–4° decomp.

$\text{B}_2\text{H}_2\text{PtCl}_6$ : orange-yellow plates +  $2\text{H}_2\text{O}$ . M.p. 257° decomp.

**Picrate**: yellow cryst. M.p. 236–8° decomp.

Willstätter, Müller, *Ber.*, 1898, **31**, 1208.

**Truxane (Bisindene)** $C_{18}H_{16}$ 

MW, 232

Plates. M.p.  $116^\circ$ . Dist.  $\rightarrow$  indene. Dist. undecomp. at low pressures.

Stobbe, Zschoch, *Ber.*, 1927, 60, 462.

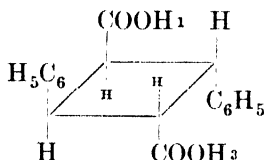
**Truxillamic Acid.**

See under Truxillic Acid.

**Truxillanic Acid.**

See under Truxillic Acid.

**$\alpha$ -Truxillic Acid** (2 : 4-Diphenylcyclobutane-1 : 3-dicarboxylic acid)

 $C_{18}H_{16}O_4$ 

MW, 296

Needles from EtOH.Aq. cryst. + 2MeOH from MeOH. M.p.  $285^\circ$ . Very sol. hot EtOH. Sol. hot AcOH. Spar. sol. Et<sub>2</sub>O, Me<sub>2</sub>CO, CS<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>, hot H<sub>2</sub>O. Sublimes in high vacuum.  $k = 4.97 \times 10^{-5}$  at  $25^\circ$ . Heat of comb. C<sub>r</sub> 2084.8 Cal., C<sub>p</sub> 2086 Cal. KOH fusion  $\rightarrow$   $\epsilon$ -truxillic acid. Dist.  $\rightarrow$  cinnamic acid. Ac<sub>2</sub>O at  $210^\circ$   $\rightarrow$   $\gamma$ -truxillic anhydride. Fuming H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  truxone. Forms very insol. Ag salt.

**Mono-Me ester** :  $C_{19}H_{18}O_4$ . MW, 310. Needles. M.p.  $195^\circ$ . Sol. C<sub>6</sub>H<sub>6</sub>, cold Na<sub>2</sub>CO<sub>3</sub>.

**Di-Me ester** :  $C_{20}H_{20}O_4$ . MW, 324. Plates or needles from MeOH. M.p.  $174^\circ$ . B.p. about  $330^\circ$ . Spar. sol. cold MeOH, AcOH.

**Mono-Et ester** :  $C_{20}H_{20}O_4$ . MW, 324. Cryst. from EtOH. M.p.  $171^\circ$ .

**Di-Et ester** :  $C_{22}H_{24}O_4$ . MW, 352. Needles. M.p.  $146^\circ$ . Spar. sol. EtOH.

**Dichloride** :  $C_{18}H_{14}O_2Cl_2$ . MW, 333. Prisms +  $\frac{1}{2}$ C<sub>6</sub>H<sub>6</sub> from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p.  $125^\circ$ . AlCl<sub>3</sub>  $\rightarrow$  truxone.

**1-Amide** :  $\alpha$ -truxillamic acid.  $C_{18}H_{17}O_3N$ . Needles from AcOH. M.p.  $261^\circ$ . Sol. EtOH, AcOH, Me<sub>2</sub>CO. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

**3-Anilide** :  $\alpha$ -truxillanic acid.  $C_{24}H_{21}O_3N$ . MW, 371. *d*-. Needles from EtOH.Aq. M.p.  $205^\circ$ .  $[\alpha]_D^{20} + 21.8^\circ$  in Me<sub>2</sub>CO. *l*-. Needles from EtOH.Aq. M.p.  $205^\circ$ .  $[\alpha]_D^{19} - 23.0^\circ$  in Me<sub>2</sub>CO. *dl*-. Needles from EtOH.Aq. M.p.

$235^\circ$ . Sol. EtOH, Me<sub>2</sub>CO, AcOEt. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

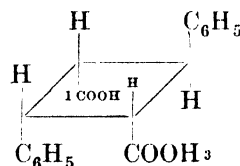
Stoermer, Stroh, *Ber.*, 1935, 68, 2108.

Stoermer, Wegner, Carl, *Ber.*, 1923, 56, 1683, 1690.

Stoermer, Foerster, *Ber.*, 1919, 52, 1256.

Kohler, *Am. Chem. J.*, 1907, 28, 238.

**$\gamma$ -Truxillic Acid** (2 : 4-Diphenylcyclobutane-1 : 3-dicarboxylic acid)

 $C_{18}H_{16}O_4$ 

MW, 296

*dl*-.

Needles from EtOH.Aq. cryst. from AcOH. M.p.  $228^\circ$ . Very sol. Et<sub>2</sub>O. Spar. sol. hot H<sub>2</sub>O.  $k = 1.08 \times 10^{-4}$  at  $25^\circ$ . Heat. at  $280^\circ$  or HCl at  $175^\circ$   $\rightarrow$   $\alpha$ -truxillic acid. Dist.  $\rightarrow$  cinnamic acid. KOH fusion  $\rightarrow$   $\alpha$ - and  $\epsilon$ -truxillic acids.

**Mono-Me ester** :  $C_{19}H_{18}O_4$ . MW, 310. Needles from EtOH.Aq. M.p.  $183.5$ – $184.0^\circ$ . Sol. EtOH, Me<sub>2</sub>CO, hot C<sub>6</sub>H<sub>6</sub>. Mod. sol. Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, ligroin, pet. ether.

**Di-Me ester** :  $C_{20}H_{20}O_4$ . MW, 324. Needles from MeOH.Aq. M.p.  $126^\circ$ . Sol. cold MeOH, AcOH.

**Mono-Et ester** :  $C_{20}H_{20}O_4$ . MW, 324. Needles from EtOH.Aq. M.p.  $173$ – $174.5^\circ$ . Very sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>.

**Di-Et ester** :  $C_{22}H_{24}O_4$ . MW, 352. Needles from EtOH.Aq. M.p.  $98^\circ$ . Sol. EtOH, Et<sub>2</sub>O, AcOH, C<sub>6</sub>H<sub>6</sub>. Spar. sol. pet. ether.

**Anhydride** :  $C_{18}H_{14}O_3$ . MW, 278. Needles from CHCl<sub>3</sub>-EtOH. M.p.  $191^\circ$  ( $180$ – $3^\circ$ ).

**Dichloride** :  $C_{18}H_{14}O_2Cl_2$ . MW, 333. Needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p.  $140^\circ$ . AlCl<sub>3</sub>  $\rightarrow$  truxone.

**3-Amide** :  $\gamma$ -truxillamic acid.  $C_{18}H_{17}O_3N$ . MW, 295. Prisms from AcOH.Aq. M.p.  $240^\circ$ . Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, pet. ether.

**Imide** :  $C_{18}H_{15}O_2N$ . MW, 277. Cryst. from AcOH or EtOH. M.p.  $208^\circ$ . Sol. C<sub>6</sub>H<sub>6</sub>. Spar. sol. Et<sub>2</sub>O. Insol. pet. ether.

**Et-imide** :  $C_{20}H_{19}O_2N$ . MW, 305. Cryst. from EtOH.Aq. M.p.  $142^\circ$ .

**Phenyl-imide** :  $C_{24}H_{19}O_2N$ . MW, 353. Cryst. from AcOH.Aq. M.p.  $194^\circ$ .

**3-Anilide** :  $\gamma$ -truxillanic acid.  $C_{24}H_{21}O_3N$ . MW, 371. Needles from EtOH.Aq. M.p.  $228^\circ$ . Sol. EtOH, Me<sub>2</sub>CO. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, ligroin.

*Mono-p-toluidide*: needles from EtOH.Aq. M.p. 268°.

*Di-p-toluidide*: needles from EtOH or AcOH. M.p. 289°.

*Di-phenylhydrazide*: m.p. 305°.

*d.*

*Mono-Me ester*: cryst. from AcOH. M.p. 145.5–147°.  $[\alpha]_D^{25} + 6.48^\circ$  in Me<sub>2</sub>CO.

*Mono-Et ester*: cryst. from AcOH. M.p. 142.5–143°.  $[\alpha]_D^{25} + 19.14^\circ$  in Me<sub>2</sub>CO.

*3-Amide*: cryst. from EtOH. M.p. 258–9°.

*3-Anilide*: needles from Et<sub>2</sub>O–ligroin. M.p. 228°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin.  $[\alpha]_D^{25} + 48.7^\circ$  in Me<sub>2</sub>CO.

*l.*

*Mono-Me ester*: needles from EtOH.Aq. M.p. 145.5–147°.

*Mono-Et ester*: cryst. from EtOH.Aq. M.p. 142.5–143°.  $[\alpha]_D^{25} - 16.48^\circ$  in Me<sub>2</sub>CO.

*3-Amide*: leaflets from AcOH.Aq. M.p. 258–9°.  $[\alpha]_D^{25} - 11.05^\circ$  in AcOH.

*3-Anilide*: needles from Et<sub>2</sub>O–ligroin. M.p. 228°.  $[\alpha]_D^{25} - 49.54^\circ$  in Me<sub>2</sub>CO.

Stoermer, Cruse, *Ber.*, 1935, **68**, 2120.

Schenk, *Ber.*, 1930, **63**, 2706.

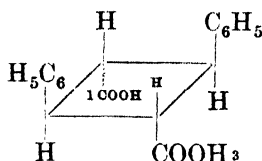
Stoermer, Fretwurst, *Ber.*, 1925, **58**, 2718.

Stoermer, Wegner, Carl, *Ber.*, 1923, **56**, 1683.

Stoermer, Emmel, *Ber.*, 1920, **53**, 497.

Lie, *Ber.*, 1889, **22**, 126.

**ε-Truxillic Acid** (2 : 4-Diphenylcyclobutane-1 : 3-dicarboxylic acid)



C<sub>18</sub>H<sub>16</sub>O<sub>4</sub> MW, 296

Needles from EtOH.Aq. or Et<sub>2</sub>O. M.p. 192°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. ligroin. Ba and Ca salts more sol. in cold than in hot H<sub>2</sub>O.

*Di-NH<sub>4</sub> salt*: cryst. Decomp. at 186°.

*Mono-Me ester*: C<sub>19</sub>H<sub>18</sub>O<sub>4</sub>. MW, 310. Prisms from MeOH. M.p. 131°.

*Di-Me ester*: C<sub>20</sub>H<sub>20</sub>O<sub>4</sub>. MW, 324. Prisms from EtOH.Aq. M.p. 64°.

*Di-Et ester*: C<sub>22</sub>H<sub>24</sub>O<sub>4</sub>. MW, 352. Prisms from EtOH. M.p. 34°.

*Dichloride*: C<sub>18</sub>H<sub>14</sub>O<sub>2</sub>Cl<sub>2</sub>. MW, 333. Prisms from ligroin. M.p. 106–7°.

*Imide*: C<sub>18</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 277. Needles from EtOH.Aq. M.p. 198°. Sol. Et<sub>2</sub>O, EtOH, Me<sub>2</sub>CO,

AcOH. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin. *K salt*: cryst. M.p. 224°.

*Et-imide*: C<sub>20</sub>H<sub>19</sub>O<sub>2</sub>N. MW, 305. Needles from EtOH. M.p. 144°.

*Phenylimide*: C<sub>24</sub>H<sub>19</sub>O<sub>2</sub>N. MW, 353. Needles from AcOH. M.p. 252°.

*3-Amide*: ε-truxillamic acid. C<sub>18</sub>H<sub>17</sub>O<sub>3</sub>N. MW, 295. Needles from EtOH. M.p. 213°. Sol. EtOH, AcOH, hot H<sub>2</sub>O. Insol. C<sub>6</sub>H<sub>6</sub>, ligroin.

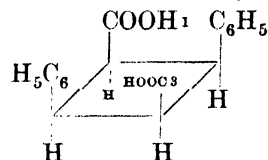
*3-Anilide*: ε-truxillanilic acid. C<sub>24</sub>H<sub>21</sub>O<sub>3</sub>N. MW, 371. Needles from EtOH. M.p. 239°.

Stoermer, Cruse, *Ber.*, 1935, **68**, 2121.

Stoermer, Neumaerker, Schmidt, *Ber.*, 1925, **58**, 2707, 2715.

Stoermer, Emmel, *Ber.*, 1920, **53**, 497.

**η-Truxillic Acid** (peri-Truxillic acid, 2 : 4-diphenylcyclobutane-1 : 3-dicarboxylic acid)



C<sub>18</sub>H<sub>16</sub>O<sub>4</sub> MW, 296

Cryst. from AcOH–C<sub>6</sub>H<sub>6</sub>. M.p. 266° (287°) decomp. Sol. EtOH. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. KOH fusion → ε-truxillic acid.

*Mono-Me ester*: C<sub>19</sub>H<sub>18</sub>O<sub>4</sub>. MW, 310. Cryst. from MeOH. M.p. 192°.

*Di-Me ester*: C<sub>20</sub>H<sub>20</sub>O<sub>4</sub>. MW, 324. Cryst. from MeOH. M.p. 104–5°.

*Anhydride*: C<sub>18</sub>H<sub>14</sub>O<sub>3</sub>. MW, 278. Prisms from AcOH. M.p. 285°.

*1-Amide*: η-truxillamic acid. C<sub>18</sub>H<sub>17</sub>O<sub>3</sub>N. MW, 295. Prisms from AcOH–C<sub>6</sub>H<sub>6</sub>. M.p. 256.5°. Spar. sol. AcOH. Insol. EtOH.

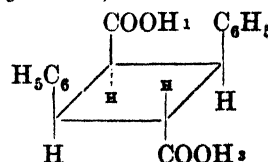
*Imide*: C<sub>18</sub>H<sub>15</sub>O<sub>2</sub>N. MW, 277. Cryst. M.p. 237°.

*1-Anilide*: η-truxillanilic acid. C<sub>24</sub>H<sub>21</sub>O<sub>3</sub>N. MW, 371. Cryst. from EtOH. M.p. 247°.

Stoermer, Möller, *Ber.*, 1935, **68**, 2131, 2132.

Stoermer, Bachér, *Ber.*, 1924, **57**, 15.

**epi-Truxillic Acid** (2 : 4-Diphenylcyclobutane-1 : 3-dicarboxylic acid)



C<sub>18</sub>H<sub>16</sub>O<sub>4</sub> MW, 296

Cryst. from EtOH.Aq. or  $C_6H_6$ -AcOH. M.p. 285-7°. Very spar. sol.  $Et_2O$ ,  $C_6H_6$ . Heat at m.p.  $\rightarrow$  ε-truxillic acid.  $Ac_2O \rightarrow$  ε-acid. KOH fusion  $\rightarrow$  ε-acid.

1-Mono-Me ester:  $C_{19}H_{18}O_4$ . MW, 310. Cryst. from MeOH. M.p. 204-5°.

3-Mono-Me ester: prisms from MeOH. M.p. 141°.

Di-Me ester:  $C_{20}H_{20}O_4$ . MW, 324. Cryst. M.p. 111-12°.

3-Amide: epi-truxillamic acid.  $C_{18}H_{17}O_3N$ . MW, 295. Cryst. M.p. 263°.

Stoermer, Möller, *Ber.*, 1935, 68, 2132.

Stoermer, Bachér, *Ber.*, 1924, 57, 15.

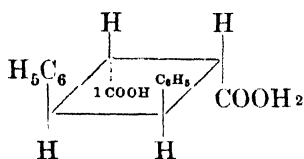
### Truxinamic Acid.

See under Truxinic Acid.

### Truxinanilic Acid.

See under Truxinic Acid.

β-Truxinic Acid (3:4-Diphenylcyclobutane-1:2-dicarboxylic acid, isotruxillic acid)



$C_{18}H_{16}O_4$

MW, 296

Cryst. from AcOH. M.p. 209-209.5°. Mod. sol. hot  $H_2O$ . KOH fusion  $\rightarrow$  δ-truxinic acid. Dist.  $\rightarrow$  cinnamic acid + stilbene. Ox.  $\rightarrow$  benzil + benzoic acid. Readily forms anhydride. Forms cryst. insol. Ca and Ba salts.

Di- $NH_4$  salt: cryst. M.p. 187°. Sol. hot EtOH.

Mono-Me ester:  $C_{19}H_{18}O_4$ . MW, 310. Cryst. from  $C_6H_6$ . M.p. 164°.

Di-Me ester:  $C_{20}H_{20}O_4$ . MW, 324. Plates from  $Et_2O$ . M.p. 76°. Very sol. EtOH,  $Et_2O$ . Heat. of comb.  $C_p$  2422.9 Cal.,  $C_v$  2421.1 Cal.

Mono-Et ester:  $C_{20}H_{20}O_4$ . MW, 324. Cryst. from  $C_6H_6$ . M.p. 133°. Sol. EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ . Insol. ligroin.

Di-Et ester:  $C_{22}H_{24}O_4$ . MW, 352. Cryst. M.p. 49-50°.

Mono-d-menthyl ester:  $C_{28}H_{34}O_4$ . MW, 434. Cryst. from EtOH. M.p. 208°. Sol.  $Me_2CO$ ,  $Et_2O$ , warm  $C_6H_6$ . Spar. sol. hot ligroin.  $[\alpha]_D^{20} - 20.9^\circ$  in  $Me_2CO$ .

Mono-l-menthyl ester: needles from EtOH. M.p. 149°. Very sol.  $Et_2O$ , EtOH,  $Me_2CO$ ,  $C_6H_6$ . Sol. hot ligroin.  $[\alpha]_D^{20} - 37.3^\circ$  in  $Me_2CO$ .

Anhydride:  $C_{18}H_{14}O_3$ . MW, 278. Prisms from  $C_6H_6$ . M.p. 116°.

Dichloride:  $C_{18}H_{14}O_2Cl_2$ . MW, 333. Plates from  $C_6H_6$ -pet. ether. M.p. 96°. Very sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. pet. ether.

2-Amide: β-truxinamic acid.  $C_{18}H_{17}O_3N$ . MW, 295. Cryst. from EtOH.Aq. M.p. 194°. Sol. EtOH,  $Me_2CO$ . Insol.  $Et_2O$ ,  $C_6H_6$ , ligroin.

Imide:  $C_{18}H_{15}O_2N$ . MW, 277. Prisms from EtOH. M.p. 224-5°. Sol. AcOH.

Phenylimide:  $C_{24}H_{19}O_2N$ . MW, 353. Cryst. M.p. 184°.

2-Anilide: β-truxinanilic acid.  $C_{24}H_{21}O_3N$ . MW, 371. dl. Cryst. from  $Me_2CO$ .Aq. M.p. 210°.

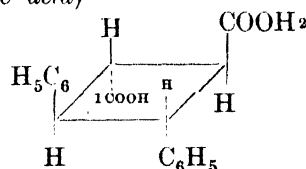
d-Menthyl ester: m.p. 171°.  $[\alpha]_D^{20} - 63^\circ$  in  $Me_2CO$ .

Stoermer, Lachmann, *Ber.*, 1926, 59, 642.

Stoermer, Laage, *Ber.*, 1921, 54, 96.

Stobbe, *Ber.*, 1919, 52, 666.

δ-Truxinic Acid (3:4-Diphenylbutane-1:2-dicarboxylic acid)



$C_{18}H_{16}O_4$

MW, 296

dl.

Cryst. from  $C_6H_6$ -AcOH, needles from hot  $H_2O$ , plates +  $1C_6H_6$  from  $C_6H_6$ . M.p. 175°. Very sol. EtOH,  $Et_2O$ ,  $CHCl_3$ . Spar. sol. boil.  $H_2O$ ,  $C_6H_6$ .

$NH_4$  salt: prisms. M.p. 206-8° decomp.

Di-Me ester:  $C_{20}H_{20}O_4$ . MW, 324. Needles from MeOH.Aq. M.p. 77°. Sol. EtOH,  $C_6H_6$ , ligroin.

Dichloride:  $C_{18}H_{14}O_2Cl_2$ . MW, 333. Cryst. from  $C_6H_6$ -ligroin. M.p. 78°. Unstable in air. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Spar. sol. ligroin.

2-Amide: δ-truxinamic acid.  $C_{18}H_{17}O_3N$ . MW, 295. M.p. 189°.

2-Anilide: δ-truxinanilic acid.  $C_{24}H_{21}O_3N$ . MW, 371. M.p. 225°.

d.

Cryst. M.p. 157-8°.  $[\alpha]_D^{20} + 8.06^\circ$  in  $Me_2CO$ . Quinine salt: needles from EtOH.Aq. M.p. 135° decomp.

l.

Needles. M.p. 158-9°.  $[\alpha]_D^{20} - 8.3^\circ$  in  $Me_2CO$ . Cinchonine salt: cryst. from EtOH.Aq. M.p. 192°.

Di-Me ester: cryst. from MeOH. M.p. 45°.  $[\alpha]_D^{20} - 11.1^\circ$  in  $Me_2CO$ .

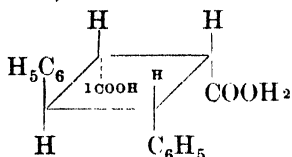


2-Amide: m.p. 206°.

Stoermer, Bachér, *Ber.*, 1922, **55**, 1860.

Stoermer, Klockmann, *Ber.*, 1925, **58**, 1164.

ζ-Truxinic Acid (3:4-Diphenylbutane-1:2-dicarboxylic acid)



$C_{18}H_{16}O_4$

MW, 296

dl.

Needles from  $Me_2CO.Aq.$  M.p. 239°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ . Spar. sol. AcOH,  $CHCl_3$ ,  $CS_2$ ,  $C_6H_6$ . Insol.  $H_2O$ , pet. ether. KOH fusion or hot HCl  $\rightarrow$  δ-truxinic acid. Forms spar. sol. Ca and Ba salts.

$NH_4$  salt: needles. Decomp. at 160°.

1-Mono-Me ester:  $C_{19}H_{18}O_4$ . MW, 310. Cryst. from MeOH. M.p. 198°. Sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ . Chloride: cryst. from  $C_6H_6$ -ligroin. M.p. 104-5°.

2-Mono-Me ester: cryst. from MeOH. M.p. 201°. Sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ . Chloride: cryst. from  $C_6H_6$ -ligroin. M.p. 120°.

Di-Me ester:  $C_{20}H_{20}O_4$ . MW, 324. Cryst. from EtOH. M.p. 116°.

Mono-Et ester:  $C_{20}H_{20}O_4$ . MW, 324. Cryst. from EtOH. M.p. 190°. Sol. EtOH,  $Et_2O$ , AcOH,  $C_6H_6$ .

Di-Et ester:  $C_{22}H_{24}O_4$ . MW, 352. Needles. M.p. 80°. Sol. EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ .

Anhydride:  $C_{18}H_{14}O_3$ . MW, 278. Cryst. from  $C_6H_6$ . M.p. 150°. Sol. EtOH,  $Me_2CO$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $Et_2O$ , pet. ether. Stable to  $H_2O$ .

Dichloride:  $C_{18}H_{14}O_2Cl_2$ . MW, 333. Prisms from  $C_6H_6$ -pet. ether. M.p. 150°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol. ligroin, pet. ether.

1-Amide: ζ-truxinamic acid.  $C_{18}H_{17}O_3N$ . MW, 295. Needles from MeOH. M.p. 222°. Sol. AcOH.

2-Amide: needles from  $Me_2CO$ . M.p. 204°.

Imide:  $C_{18}H_{15}O_2N$ . MW, 277. Needles from EtOH. M.p. 168-168.5°. Sol. hot  $Na_2CO_3$ .

Phenylimide:  $C_{24}H_{19}O_2N$ . MW, 353. Needles from EtOH. M.p. 180°.

1-Anilide: ζ-truxinanilic acid.  $C_{24}H_{21}O_3N$ . MW, 371. Cryst. from epichlorohydrin. M.p. 237°.

2-Anilide: needles from EtOH.Aq. M.p. 214°.

d.

Cryst. from  $Me_2CO$ . M.p. 222°.  $[\alpha]_D^{20} + 65.42$  in EtOH.

Di-Me ester: cryst. from MeOH. M.p. 106°.  $[\alpha]_D + 89.88^\circ$  in  $Me_2CO$ .

l.

Cryst. from  $Me_2CO$ . M.p. 222°.  $[\alpha]_D^{20} - 78.37^\circ$  in EtOH.

Cinchonine salt: needles from EtOH. M.p. 192°.

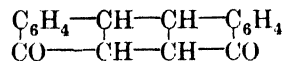
Anhydride: cryst. M.p. 162°.  $[\alpha]_D - 145.84^\circ$  in  $Me_2CO$ .

Dichloride: cryst. M.p. 160°.  $[\alpha]_D - 98.6^\circ$  in  $C_6H_6$ .

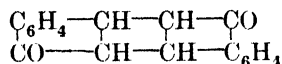
Stoermer, Klockmann, *Ber.*, 1925, **58**, 1164.

Stoermer, Scholtz, *Ber.*, 1921, **54**, 85.

### α-Truxone



or



$C_{18}H_{12}O_2$

MW, 260

Plates from AcOH, powder from  $C_6H_6$  needles from dil.  $HNO_3$ . M.p. 294°. Spar. sol. most org. solvents. Sublimes. Very stable to oxidising agents.

Dioxime: needles. Does not melt below 300°. Spar. sol. most org. solvents. Di-Me ether: cryst. from EtOH. M.p. 214°. Diacetyl: needles. M.p. 261°.

Anil: needles. M.p. 270° decomp.

Phenylhydrazone: yellow needles. M.p. 270°.

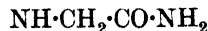
Stobbe, Zschoch, *Ber.*, 1927, **60**, 470.

Stoermer, Foerster, *Ber.*, 1919, **52**, 1255.

### Trypaflavine.

See under 2: 8-Diaminoacridine.

### Tryparsamide

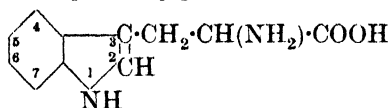


$C_8H_9O_4N_2Na_2As$

MW, 318

Cryst. +  $3H_2O$ . Employed in treatment of trypanosome and spirochaete infections. Low toxicity.

Jacobs, Heidelberger, *Organic Syntheses*, Collective Vol. I, 475.

**Tryptamine.**3-[ $\omega$ -Aminoethyl]-indole, *q.v.***Tryptophane** (1-Amino-2-[3-indolyl]-propionic acid, 2-[3-indolyl]- $\alpha$ -alanine) $C_{11}H_{12}O_2N_2$ 

MW, 204

*l.*

Constituent of many plants. Enzymatic hyd. product of most plant and animal proteins. Plates from EtOH.Aq. M.p. 278° (252°, 289°). Sol. boiling  $H_2O$ , hot EtOH. Spar. sol. cold  $H_2O$ , EtOH. Insol.  $CHCl_3$ . Aq. sol. shows acid reaction.  $[\alpha]_D^{20} - 33.4^\circ$  in EtOH,  $[\alpha]_D^{20} + 6.1^\circ$  in  $N/NaOH$ . Heat with Py or 25% HCl at 170°  $\rightarrow$  *dl.*  $FeCl_3 \rightarrow$  indole-3-aldehyde. Br water  $\rightarrow$  reddish-violet col. KOH fusion  $\rightarrow$  skatole + oxalic acid + glyoxylic acid.

*B,HCl*: needles from MeOH. M.p. 257° decomp.

*Picrate*: red needles and plates. M.p. 195–6° slight decomp.

*Picrolonate*: orange-red needles from  $H_2O$ . M.p. 203–4° decomp.

*Me ester*:  $C_{12}H_{14}O_2N_2$ . MW, 218. Plates from  $Et_2O$ . M.p. 89–5°. Very sol. MeOH. Sol.  $Et_2O$ , AcOEt. Spar. sol. pet. ether. *B,HCl*: microneedles from MeOH or AcOEt. M.p. 214° decomp.

*Chloride*:  $C_{11}H_{11}ON_2Cl$ . MW, 222.5. *B,HCl*: darkens at 172°, sinters at 208°, m.p. 228° decomp.

*Amide*:  $C_{11}H_{13}ON_3$ . MW, 203. M.p. 167–70°.  $[\alpha]_D^{20} - 7.9^\circ$  in EtOH.

*Et-amide*:  $C_{13}H_{17}ON_3$ . MW, 231. M.p. 67–9°.  $[\alpha]_D^{20} - 14.4^\circ$  in EtOH.

*Di-Et amide*:  $C_{15}H_{21}ON_3$ . MW, 259. M.p. 183–5°.  $[\alpha]_D^{20} - 24.7^\circ$  in EtOH.

*Anilide*: m.p. 83–5°.  $[\alpha]_D^{20} - 9.5^\circ$  in EtOH.

*Et-anilide*: m.p. 97–9°.  $[\alpha]_D^{20} - 4.2^\circ$  in EtOH.

*N-Chloroacetyl*: plates from  $H_2O$ . M.p. 159°.

*N-Benzenesulphonyl*: cryst. from EtOH.Aq. M.p. 185° decomp.

*dl.*

Plates from EtOH.Aq. M.p. 275–82°.

$\alpha$ -*N-Me*:  $C_{12}H_{14}O_2N_2$ . MW, 218. Needles from EtOH.Aq. Darkens at 280°, m.p. 297° decomp. *Picrate*: plates from MeOH–pet. ether. M.p. 186° decomp.

$\alpha$ -*N-Di-Me*:  $C_{13}H_{16}O_2N_2$ . MW, 232. Needles from  $Et_2O$ –pet. ether. M.p. 49–50°. *Methiodide*:

Dict. of Org. Comp.—III.

needles from MeOH. M.p. 210–11°. *Picrate*: prisms from MeOH. M.p. 170–1°.

*N-3:5-Dinitrobenzoyl*: m.p. 240°.

*N-Benzenesulphonyl*: m.p. 185° decomp.

*N-p-Toluenesulphonyl*: needles from  $Me_2CO$ . M.p. 176°.

Boyd, Robson, *Biochem. J.*, 1935, **29**, 2256.

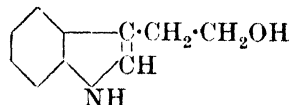
Gordon, Jackson, *J. Biol. Chem.*, 1935, **110**, 151.

Hoshino, Shimodaira, *Ann.*, 1935, **520**, 25.

Bauguess, Berg, *J. Biol. Chem.*, 1934, **106**, 618.

Cox, King, *Organic Syntheses*, 1930, X, 100.

**Tryptophol** (2-[3-Indolyl]-ethyl alcohol, 3- $\omega$ -hydroxyethylindole)

 $C_{10}H_{11}ON$ 

MW, 161

Prisms from  $C_6H_6$ –pet. ether, plates from  $Et_2O$ –pet. ether. M.p. 59°. Sol. MeOH, EtOH,  $Et_2O$ ,  $Me_2CO$ ,  $CHCl_3$ , AcOH, AcOEt. Spar. sol. cold  $H_2O$ , ligroin, pet. ether. Heat or boil. with alkalis  $\rightarrow$  indole. Sol. warm conc.  $H_2SO_4 \rightarrow$  red col.

*N-Benzoyl*: yellow prisms and plates from ligroin. M.p. 76°.

*Picrate*: red needles from  $H_2O$ . M.p. 100–1°.

*Phenylurethane*: m.p. 130–1°.

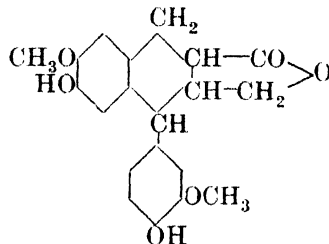
Jackson, *J. Biol. Chem.*, 1930, **88**, 659.

Ehrlich, *Ber.*, 1912, **45**, 884.

**Tsugalactone.**

See Tsugaresinol.

**Tsugaresinol** (*Tsugalactone*, *condendrin*, *sulphite liquors lactone*)

 $C_{20}H_{20}O_6$ 

MW, 356

Constituent of spruce resin and of the wood of *Tsuga Sieboldii*. Found in sulphite waste liquors from wood pulp manufacture. Cryst. from EtOH. M.p. 254–5°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Insol. pet. ether.  $[\alpha]_D^{20} - 54.5^\circ$  in  $Me_2CO$ . Alc.  $FeCl_3 \rightarrow$  green col. Conc.

$\text{H}_2\text{SO}_4 + \text{NaNO}_2 \longrightarrow \text{brown} \longrightarrow \text{red} \longrightarrow \text{blue col.}$

*Di-Me ether*:  $\text{C}_{22}\text{H}_{24}\text{O}_6$ . MW, 384. M.p. 178.5–179°.  $[\alpha]_D^{18} - 99.4^\circ$  in  $\text{Me}_2\text{CO}$ .

*Di-Et ether*:  $\text{C}_{24}\text{H}_{28}\text{O}_6$ . MW, 412. Needles from EtOH. M.p. 178–9°.

*Diacetyl*: cryst. from EtOH– $\text{Me}_2\text{CO}$ . M.p. 204–5° (222°).  $[\alpha]_D^{18} - 68.4^\circ$  in  $\text{Me}_2\text{CO}$ .

*Di-p-nitrobenzoyl*: prisms from  $\text{CHCl}_3$ –Et<sub>2</sub>O. M.p. 257–8°.

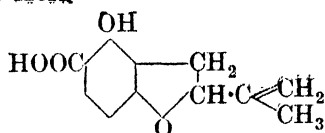
Emde, Schartner, *Helv. Chim. Acta*, 1935, **18**, 344.

Haworth, Sheldrick, *J. Chem. Soc.*, 1935, 636.

Kawamura, *Chem. Zentr.*, 1932, II, 60.

Holmberg, *Ber.*, 1921, **54**, 2406.

### Tubaic Acid



$\text{C}_{12}\text{H}_{12}\text{O}_4$  MW, 220

Needles from Et<sub>2</sub>O–pet. ether. M.p. 129°.  $[\alpha]_D^{20} - 73.0^\circ$  in  $\text{CHCl}_3$ . Loses  $\text{CO}_2$  at 185–200°.  $\text{FeCl}_3 \longrightarrow \text{red col.}$

*Me ester*:  $\text{C}_{13}\text{H}_{14}\text{O}_4$ . MW, 234. Needles from MeOH.Aq. M.p. 52°.  $[\alpha]_D^{19} - 72.8^\circ$  in  $\text{CHCl}_3$ .  $\text{FeCl}_3 \longrightarrow \text{red col.}$

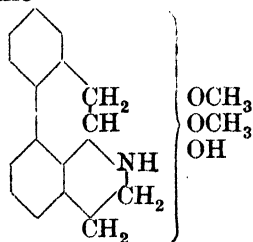
*Me ether*:  $\text{C}_{13}\text{H}_{14}\text{O}_4$ . MW, 234. Cryst. from EtOH.Aq. M.p. 78°. *Me ester*:  $\text{C}_{14}\text{H}_{16}\text{O}_4$ . MW, 248. Oil. B.p. 175°/4.5 mm.

*Acetyl*: m.p. 133°.

Haller, La Forge, *J. Am. Chem. Soc.*, 1932, **54**, 1988; 1930, **52**, 3211.

Takei, Koide, *Ber.*, 1929, **62**, 3032.

### Tuduranine



Suggested constitution

$\text{C}_{18}\text{H}_{19}\text{O}_3\text{N}$  MW, 297

Alkaloid from *Sinomenium acutum*. Poorly cryst. M.p. 105–25°. Sol. most org. solvents. Sol. alkalis.  $\text{FeCl}_3 \longrightarrow \text{faint col.}$   $\text{H}\cdot\text{CHO} + \text{H}_2\text{SO}_4 \longrightarrow \text{fuchsin-red col.}$

*B,HCl*: prisms from  $\text{H}_2\text{O}$ . M.p. 286° decomp.  $[\alpha]_D^{14.5} - 148^\circ$  in MeOH.Aq.

*Me ether*:  $\text{C}_{19}\text{H}_{21}\text{O}_3\text{N}$ . MW, 311. N-Acetyl:

prisms from EtOH. M.p. 189°.  $[\alpha]_D^{18} - 400-17^\circ$  in  $\text{CHCl}_3$ .

*N-Me*:  $\text{C}_{19}\text{H}_{21}\text{O}_3\text{N}$ . MW, 311. *Methiodide*: cryst. from EtOH. M.p. 224°.

*N-Et-O-Et*:  $\text{C}_{22}\text{H}_{27}\text{O}_3\text{N}$ . MW, 353. *Ethiodide*: M.p. 238°.  $[\alpha]_D^{18} - 112.47^\circ$  in  $\text{CHCl}_3$ .

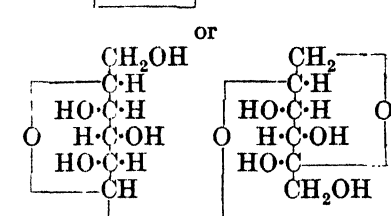
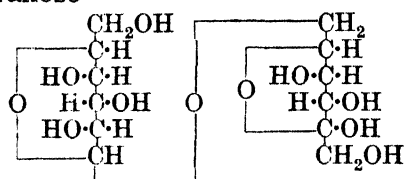
*N-Di-Et-O-Et*:  $\text{C}_{24}\text{H}_{31}\text{O}_3\text{N}$ . MW, 381. *Ethiodide*: m.p. 163–4°.

*N-Acetyl*: prisms from MeOH. M.p. 277°.  $[\alpha]_D^{18} - 395.24^\circ$  in MeOH– $\text{CHCl}_3$ .

*O*: N-Diacetyl: prisms from MeOH or EtOH. M.p. 170°.  $[\alpha]_D^{18} - 321.7^\circ$  in MeOH.

Goto, *Ann.*, 1935, **521**, 175.

### Turanose



$\text{C}_{12}\text{H}_{22}\text{O}_{11}$  MW, 342

Amorphous powder, or prisms +  $\frac{1}{4}$  MeOH from MeOH. M.p. 65–70° (60–5°), MeOH free 157°. Sol.  $\text{H}_2\text{O}$ , MeOH. Less sol. EtOH. Sweet taste.  $[\alpha]_D^{20} + 65-8^\circ$  in  $\text{H}_2\text{O}$ , ( $[\alpha]_D^{20} + 22.0^\circ \longrightarrow +75.3^\circ$  in  $\text{H}_2\text{O}$ ). Reduces Fehling's. Hyd. by dil. acids or by yeast  $\longrightarrow$  glucose + fructose.

*Phenylosazone*: yellow needles from dil. EtOH. M.p. 215–20° decomp. Sol. hot  $\text{H}_2\text{O}$ .

*Hepta-Me ether*: yellow syrup. B.p. 185–90°/0.2 mm., 162–3°/0.06 mm.  $[\alpha]_D^{19} + 106.0^\circ \longrightarrow 104.8^\circ$  in EtOH.  $n_D 1.4652$ . *Me glucoside*: yellow syrup. B.p. 159–62°/0.15 mm.  $[\alpha]_D^{19} + 106.7^\circ$  in  $\text{H}_2\text{O}$ , + 109.7° in EtOH.

*Hepta-acetyl*: (i) needles from Et<sub>2</sub>O. M.p. 140–1°.  $[\alpha]_D^{20} + 37^\circ$  in  $\text{CHCl}_3$ . (ii) Prisms. M.p. 147°.  $[\alpha]_D^{18} + 38.7^\circ \longrightarrow 41.7^\circ$  in  $\text{CHCl}_3$ .

Fischer, *Ber.*, 1894, **27**, 2488.

Zemplén, Braun, *Ber.*, 1926, **59**, 2230.

Zemplén, *ibid.*, 2539.

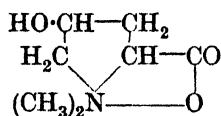
Leitch, *J. Chem. Soc.*, 1927, 588.

Åagaard, *Chem. Abstracts*, 1930, **24**, 1089.

Pacsu, *J. Am. Chem. Soc.*, 1931, **53**, 3099; 1933, **55**, 2451.

Hudson, Pacsu, *J. Am. Chem. Soc.*, 1930, **52**, 2519.

## Turicine

C<sub>7</sub>H<sub>13</sub>O<sub>3</sub>N

MW, 159

Constituent of *Betonica wiesen* and *Stachys silvatica*. Stereoisomeric with betonicine. Needles or prisms + 1H<sub>2</sub>O from EtOH.Aq. M.p. anhyd. 260° decomp. Very sol. H<sub>2</sub>O. [α]<sub>D</sub> + 41.5° (anhyd.) in H<sub>2</sub>O. Pptd. from aq. sol. by phosphotungstic acid.

*B,HCl*: needles or plates from EtOH. Decomp. at 224°. [α]<sub>D</sub> + 25.7° in H<sub>2</sub>O.

*B,HAuCl<sub>4</sub>*: yellow prisms. Decomp. at 230–2°.

*B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub>,H<sub>2</sub>O*: m.p. 223° decomp.

Goodson, Clewer, *J. Chem. Soc.*, 1919, 115, 931.

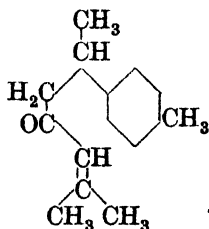
Küng, Trier, *Z. physiol. Chem.*, 1913, 85, 209.

Turmerone (*Tumerone*)C<sub>15</sub>H<sub>22</sub>O

MW, 218

Occurs in essential oil from *Curcuma longa*. Oil. B.p. 125–6°/10 mm.

Rupe, Clar, Pfau, Plattner, *Helv. Chim. Acta*, 1934, 17, 372.

*ar*-TurmeroneC<sub>15</sub>H<sub>20</sub>O

MW, 216

Occurs in essential oil from *Curcuma longa*. Pale yellow oil. B.p. 159–60°/10 mm. D<sub>20</sub> 0.9571. n<sub>D</sub><sup>20</sup> 1.5219. [α]<sub>D</sub><sup>20</sup> + 82.21°.

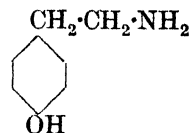
*Oxime*: oil. B.p. 179–80°/10 mm.

*Semicarbazone*: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 108–9°.

*2:4-Dinitrophenylhydrazone*: orange-yellow needles. M.p. 133–4°.

Rupe, Gassmann, *Helv. Chim. Acta*, 1936, 19, 569.

See also previous reference.

Tyramine (*Tyrosamine*, 2-*p*-hydroxyphenylethylamine, *p*-β-aminoethylphenol)C<sub>8</sub>H<sub>11</sub>ON

MW, 137

Chief pressor base found in some extracts of ergot, putrified animal tissues, mature cheese, mistletoes. Cryst. from EtOH or anisole, plates or needles from C<sub>6</sub>H<sub>6</sub>. M.p. 164–164.5°. B.p. 205–7°/25 mm., 195°/13 mm., 165–7°/2 mm. Spar. sol. xylene. Sol. 95 parts H<sub>2</sub>O at 15°. Sol. 8 parts boiling EtOH. Aq. sol. reacts alkaline. KOH fusion → *p*-hydroxybenzoic acid. Hydrochloride + KNO<sub>3</sub> in neutral sol. → tyrosol.

*B,HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 269°.

*Oxalate*: m.p. 203–4°.

*Picrate*: prisms. M.p. 206°.

*Me ether*: C<sub>9</sub>H<sub>13</sub>ON. MW, 151. Oil with fish-like odour. B.p. 138–40°/20 mm., 132–4°/14 mm. Very spar. sol. H<sub>2</sub>O. Absorbs CO<sub>2</sub> from the air. *B,HCl*: plates from EtOH. M.p. 271–2°. *N*-Benzenesulphonyl: cryst. from EtOH or C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 79–80°.

*N*-*Me*: C<sub>9</sub>H<sub>13</sub>ON. MW, 151. Prisms from EtOH, plates from C<sub>6</sub>H<sub>6</sub>. M.p. 130°. B.p. 183–5°/9 mm. Spar. sol. H<sub>2</sub>O. *B,HCl*: plates or needles from EtOH-Et<sub>2</sub>O. M.p. 148–5°. *B<sub>1</sub>(COOH)<sub>2</sub>*: needles from EtOH. M.p. 250° decomp. *B<sub>2</sub>,H<sub>2</sub>PtCl<sub>6</sub>*: yellow needles. M.p. 205°. *Picrate*: m.p. 149°. *Picolonate*: m.p. 234–5°. *N*-Acetyl: needles from H<sub>2</sub>O, plates from EtOH or AcOEt. M.p. 142°. *N*-Benzenesulphonyl: plates from EtOH-Et<sub>2</sub>O. M.p. 133–5°. *Dibenzoyl*: prisms from pet. ether. M.p. 99°.

*N*-*Di*-*Me*: see Hordenine.

*N*-*Et*: C<sub>10</sub>H<sub>15</sub>ON. MW, 165. Needles from EtOH. M.p. 157–8°. B.p. 185–7°/9 mm. *B,HCl*: pale red needles from EtOH-Et<sub>2</sub>O. M.p. 184–5°. *B<sub>1</sub>(COOH)<sub>2</sub>*: plates from EtOH. M.p. 245° decomp. *Picolonate*: m.p. 216° decomp.

*N*-Chloroacetyl: cryst. M.p. 109°.

*N*-Benzoyl: plates from EtOH. M.p. 162°.

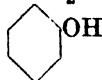
*O*: *N*-Dibenzoyl: needles from EtOH.Aq. M.p. 172°.

Buck, *J. Am. Chem. Soc.*, 1933, 55, 3389.

Waser, *Helv. Chim. Acta*, 1925, 8, 766.

Barger, *J. Chem. Soc.*, 1909, 95, 1127, 1722.

**o-Tyrosine** (2-o-Hydroxyphenyl- $\alpha$ -alanine, 1-amino-2-o-hydroxyphenyl-propionic acid, 2-hydroxy- $\alpha$ -aminohydrocinnamic acid)



$\text{C}_9\text{H}_{11}\text{O}_3\text{N}$

MW, 181

Needles from  $\text{H}_2\text{O}$ , plates from EtOH.Aq. M.p. 249–50°. Sol. 500 parts  $\text{H}_2\text{O}$  at 17°. Sol. warm AcOH. Insol.  $\text{Et}_2\text{O}$ .  $\text{FeCl}_3 \rightarrow$  red  $\rightarrow$  violet col.

*B.HCl*: prisms. Decomp. at 180°.

*Me ester*:  $\text{C}_{10}\text{H}_{13}\text{O}_3\text{N}$ . MW, 195. *B.HCl*: m.p. 179°.

*Anhydride*: diacetyl, needles from EtOH. M.p. 225°.

*Me ether*:  $\text{C}_{10}\text{H}_{13}\text{O}_3\text{N}$ . MW, 195. Needles from  $\text{H}_2\text{O}$ . M.p. 206° decomp.

*N-Benzoyl*: cryst. from  $\text{H}_2\text{O}$ . M.p. 176°.

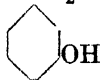
*Dibenzoyl*: cryst. from  $\text{C}_6\text{H}_6$ . M.p. 172°.

Dickinson, Marshall, *J. Chem. Soc.*, 1929, 1497.

Ueda, *Chem. Zentr.*, 1928, I, 2618.

Johnson, Scott, *J. Am. Chem. Soc.*, 1915, 37, 1853.

**m-Tyrosine** (2-m-Hydroxyphenyl- $\alpha$ -alanine, 1-amino-2-m-hydroxyphenyl-propionic acid, 3-hydroxy- $\alpha$ -aminohydrocinnamic acid)



$\text{C}_9\text{H}_{11}\text{O}_3\text{N}$

MW, 181

Needles or plates from  $\text{H}_2\text{O}$ , plates from EtOH.Aq. M.p. 275° (280°). Sol. 120 parts  $\text{H}_2\text{O}$  at 17°, 22 parts at 100°. Sol. cold AcOH.Aq. Spar. sol. EtOH.

*Anhydride*: cryst. from EtOH.Aq. M.p. 276–7°. *Diacetyl*: needles from EtOH. M.p. 189–90°.

*N-Benzoyl*: cryst. from  $\text{H}_2\text{O}$ . M.p. 180°.

See first two references above and also Blum, *Chem. Zentr.*, 1908, II, 1946.

**Tyrosine** (p-Tyrosine, 2-p-hydroxyphenyl- $\alpha$ -alanine, 1-amino-2-p-hydroxyphenyl-propionic acid, 4-hydroxy- $\alpha$ -aminohydrocinnamic acid)



$\text{C}_9\text{H}_{11}\text{O}_3\text{N}$

MW, 181

d-.

Cryst. Melts with decomp.  $[\alpha]_D^{20} + 8.64^\circ$  in 21% HCl. Aq. sol. + tyrosinase  $\rightarrow$  red col.

*N-Benzoyl*: m.p. 165.5°.  $[\alpha]_D^{20} - 19.59^\circ$  in alk. sol.

l-.

Widely distributed in plant and animal proteins. Needles from  $\text{H}_2\text{O}$ . Decomp. (slow heat.) at 290–5°, (rapid heat.) at 314–18°. Triboluminescent. Sol. 2491 parts  $\text{H}_2\text{O}$  at 17°, 13,500 parts cold 96% EtOH, 700 parts AcOH at 16°. Sol. acids, alkalis, ammonia. Insol.  $\text{Et}_2\text{O}$ .  $[\alpha]_D^{20} - 8.07^\circ$  in 21% HCl,  $-9.01^\circ$  in 11.6% KOH. Heat of comb.  $C_p$  1071.2 Cal.,  $C_v$  1070.8 Cal.  $k = 4 \times 10^{-9}$  at 25°. Heat at 270°  $\rightarrow$  tyramine. Stable to saturated HCl and HBr at 240°. NaOH fusion  $\rightarrow$  p-hydroxybenzoic acid. Heat with  $\text{Ba}(\text{OH})_2$ . Aq. at 170°  $\rightarrow$  dl-form. Boiling dil. AcOH sol. +  $\text{NaNO}_2 \rightarrow$  violet or red col. HCl sol. + Cl water +  $\text{NH}_3 \rightarrow$  red col. Tyrosinase  $\rightarrow$  red  $\rightarrow$  black "melanin" pigment. Warm  $\text{HNO}_3 \rightarrow$  yellow col.  $\text{TiO}_2$  in  $\text{H}_2\text{SO}_4 \rightarrow$  dark orange-yellow col. Paraformaldehyde + pure conc.  $\text{H}_2\text{SO}_4 \rightarrow$  green col. Alloxan in  $\text{H}_2\text{O} \rightarrow$  murexide col. Forms cryst. salts. Ba and Ag salts spar. sol. cold  $\text{H}_2\text{O}$ .

*Cu salt*: dark blue needles. Spar. sol. cold  $\text{H}_2\text{O}$ . Sol. hot  $\text{H}_2\text{O}$ .

*Picrolonate*: decomp. at 260°.

*Me ester*:  $\text{C}_{10}\text{H}_{13}\text{O}_3\text{N}$ . MW, 195. Prisms from AcOEt. M.p. 135–6°. Very sol. MeOH. Sol. hot  $\text{H}_2\text{O}$ , EtOH, AcOEt, alkalis. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ .  $[\alpha]_D^{20} + 25.75^\circ$  in MeOH. *N-Me*:  $\text{C}_{11}\text{H}_{15}\text{O}_3\text{N}$ . MW, 209. Prisms from AcOEt. M.p. 116–17°. Spar. sol.  $\text{Me}_2\text{CO}$ .

*Et ester*:  $\text{C}_{11}\text{H}_{15}\text{O}_3\text{N}$ . MW, 209. Prisms from AcOEt. M.p. 108–9°. Sol. EtOH, hot  $\text{C}_6\text{H}_6$ , AcOEt. Spar. sol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ .  $[\alpha]_D^{20} + 20.4^\circ$  in EtOH. *Hydrochloride*: needles from EtOH- $\text{Et}_2\text{O}$  or AcOEt. M.p. 166°. *N-Chloroacetyl*: needles from  $\text{CHCl}_3$ -pet. ether. M.p. 88–9°. *N-Bromoacetyl*: m.p. 101°. *N-Iodoacetyl*: plates from  $\text{C}_6\text{H}_6$ . M.p. 120°. *N-p-Toluenesulphonyl*: needles from  $\text{CHCl}_3$ -pet. ether. M.p. 114°.

*Amide*:  $\text{C}_9\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 180. Prisms or plates from EtOH. M.p. 153–4°. Sol.  $\text{H}_2\text{O}$ , EtOH.  $[\alpha]_D^{20} + 19.47^\circ$  in  $\text{H}_2\text{O}$ .

*Anhydride*:  $(\text{C}_9\text{H}_9\text{O}_2\text{N})_n$ . Two forms. ( $\alpha$ -) Needles from EtOH. M.p. 278–9°. Spar. sol. cold  $\text{H}_2\text{O}$ , cold EtOH. Cryst. from EtOH repeatedly  $\rightarrow$   $\beta$ - ( $\beta$ -) Greyish powder. M.p. 279° slight decomp.

*Hydrazide*: m.p. 195.5°.

N-Me: see Surinamine.

Me ether: plates. M.p. 264–5°.  $[\alpha]_{D}^{20} -5.9^\circ$  in HCl.  $B, H_2SO_4$ : m.p. 191°.  $B, HCl$ : needles from dil. HCl. M.p. 237–8° decomp. Picrolonate: needles. Softens at 145°, m.p. 174° decomp. N-Acetyl: plates from  $H_2O$ . M.p. 150–1°. N-Benzoyl: needles from  $H_2O$ . M.p. 136–7°.

N-Formyl: prisms or plates +  $1H_2O$  from  $H_2O$ . Anhyd. in vacuo at 100°. M.p. anhyd. 171–4° decomp.  $[\alpha]_{D}^{20} +84.8^\circ$  in EtOH.

N-Acetyl: cryst. from  $H_2O$ . Plates from dioxan. M.p. 146–8°.

N-Chloroacetyl: prisms from  $H_2O$ . M.p. 155–6°.

N-Benzoyl: plates from  $H_2O$ . M.p. 165–6°.

N-p-Toluenesulphonyl: needles or prisms from EtOH.Aq. M.p. 187–8°.

O: N-Diacetyl: cryst. M.p. 172°.

O: N-Dibenzoyl: needles from AcOH. M.p. 211–12°.

dl-.

Occurs naturally. Plates or needles from  $H_2O$ . Decomp. (slow heat.) at 290–5°, (rapid heat.) at 340°. Sol. 2454 parts  $H_2O$  at 20°, 154 at 100°. Insol. cold EtOH,  $Et_2O$ .

Et ester:  $B, HCl$ : m.p. 166°. O: N-Diacetyl: m.p. 90°. N-Benzoyl: needles from EtOH.Aq. or  $C_6H_6$ -ligroin. M.p. 122–3°.

Isoamyl ester:  $C_{14}H_{21}O_3N$ . MW, 251. Yellow needles. M.p. 68–70°. Sol. EtOH, AcOEt,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $H_2O$ .  $B, HCl$ : needles from AcOEt- $Et_2O$ . M.p. 181–2°. N-Benzoyl: needles. M.p. 106–7°.

Amide: N-benzoyl, needles from EtOH.Aq. M.p. 232–3°.

Anhydride:  $(C_9H_9O_2N)_n$ . Two forms. (α-) Needles from EtOH. M.p. 278–9°. (β-) Amorph. M.p. 279°.

Hydrazide: needles from EtOH. M.p. 171°. N-Benzoyl: needles. M.p. 229–30°.

Me ether: plates or prisms. M.p. about 295° decomp. Sol. HCl,  $NH_3$ . Spar. sol.  $H_2O$ , hot AcOH.

N-Me: needles. Decomp. at 318°.

O: N-Di-Me:  $C_{11}H_{15}O_3N$ . MW, 209. Needles from  $H_2O$ . Decomp. at 220–55°.

N-Benzoyl: needles from AcOH or hot  $H_2O$ . M.p. 191–3°. Anilide: microcryst. M.p. 212°.

p-Toluenesulphonyl: columns. M.p. 224–6°.

Bucherer, Lieb, *J. prakt. Chem.*, 1934, 141, 37.

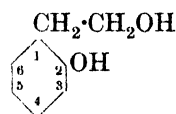
Chikano, *Z. physiol. Chem.*, 1929, 180, 249.

Curtius, Donselt, *J. prakt. Chem.*, 1917, 95, 349.

Mörner, *Z. physiol. Chem.*, 1913, 88, 126.

Fischer, *Ber.*, 1901, 34, 451; 1899, 32, 3644.

o-Tyrosol (o-β-Hydroxyethylphenol, o-hydroxyphenylethyl alcohol)



$C_8H_{10}O_2$

MW, 138

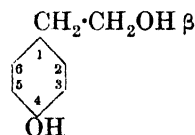
Oil. B.p. 168–9°/12 mm. Spar. misc. with cold  $H_2O$ .  $D^{18}_4$  1.1531.  $n_D^{18}$  1.5575.  $FeCl_3 \rightarrow$  blue col.

2-Et ether:  $C_{10}H_{14}O_2$ . MW, 166. Oil. B.p. 136–7°.

Mono-acetyl deriv.: cryst. from ligroin. M.p. 64.5°. B.p. 170–80°/30 mm.

Stoermer, Kahlert, *Ber.*, 1901, 34, 1809.

Tyrosol (p-Hydroxyphenylethyl alcohol, p-β-hydroxyethylphenol, p-tyrosol)



$C_8H_{10}O_2$

MW, 138

Needles from  $CHCl_3$ . M.p. 93°. B.p. about 310°, 195°/18 mm. Very sol.  $H_2O$ , EtOH,  $Et_2O$ ,  $Me_2CO$ , AcOH. Sol.  $CHCl_3$ ,  $C_6H_6$ ,  $CS_2$ . Spar. sol. pet. ether.  $FeCl_3 \rightarrow$  blue col. Hot conc.  $H_2SO_4 \rightarrow$  red col.  $H \cdot CHO + H_2SO_4 \rightarrow$  yellowish-green sol.  $\rightarrow$  green ppt. with  $H_2O$ .

4-Me ether:  $C_9H_{12}O_2$ . MW, 152. Plates. M.p. 24°. B.p. 143°/13 mm. Insol. ligroin. Acetyl: b.p. 277–8°, 156–7°/11 mm.  $D^0$  1.101.

4-Et ether:  $C_{10}H_{14}O_2$ . MW, 166. M.p. about 40°. B.p. 135–40°/7 mm.

β-Acetyl: prisms from  $Et_2O$ -ligroin. M.p. 59°. B.p. 192°/18 mm.

Diacetyl: oil. B.p. 187°/18 mm.

Dibenzoyl: needles from EtOH. M.p. 111°.

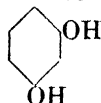
v. Braun, *Ber.*, 1912, 45, 1283.

Neubauer, Fromherz, *Z. physiol. Chem.*, 1910, 70, 342.

## U

**Ulexine.**

See Cytisine.

**Umbellic Acid** (2:4-Dihydroxycinnamic acid)

MW, 180

Yellow powder. Darkens at  $240^\circ$ , decomp. at  $260^\circ$ . Sol. EtOH, warm  $\text{H}_2\text{O}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ , ligroin. Readily decomp.  $k = 1.88 \times 10^{-5}$  at  $25^\circ$ . Pb and Cu salts insol.  $\text{H}_2\text{O}$ .

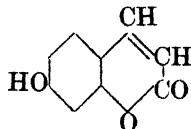
*Me ester*:  $\text{C}_{10}\text{H}_{10}\text{O}_4$ . MW, 194. *Di-Me ether*:  $\text{C}_{12}\text{H}_{14}\text{O}_4$ . MW, 222. Needles from EtOH.Aq. M.p.  $87^\circ$ . Distils above  $360^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. ligroin.

*Et ester*:  $\text{C}_{11}\text{H}_{12}\text{O}_4$ . MW, 208. *Di-Me ether*:  $\text{C}_{13}\text{H}_{16}\text{O}_4$ . MW, 236. Needles from pet. ether. M.p.  $61^\circ$ . B.p.  $208-12^\circ/13$  mm. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. pet. ether.

*4-Me ether*:  $\text{C}_{10}\text{H}_{10}\text{O}_4$ . MW, 194. Needles from  $\text{H}_2\text{O}$ . M.p.  $180-5^\circ$  decomp.

*Di-Me ether*:  $\text{C}_{11}\text{H}_{12}\text{O}_4$ . MW, 208. Two forms. ( $\alpha$ -) Needles from EtOH. M.p.  $138^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Heat. alone or with  $\text{HCl} \rightarrow \beta$ -form. ( $\beta$ -) Needles from  $\text{H}_2\text{O}$  or EtOH.Aq. M.p.  $186^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ . Insol. ligroin. Sol.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow col. Sublimes.

*Di-Et ether*:  $\text{C}_{13}\text{H}_{16}\text{O}_4$ . MW, 236. Two forms. ( $\alpha$ -) Plates from EtOH.Aq. M.p.  $106.5^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol.  $\text{H}_2\text{O}$ . Heat  $\rightarrow \beta$ -form with part. decomp. ( $\beta$ -) Cryst. from EtOH. M.p.  $200^\circ$ . Sol.  $\text{Et}_2\text{O}$ . Spar. sol.  $\text{H}_2\text{O}$ .

Will, Beck, *Ber.*, 1886, 19, 1778.Posen, *Ber.*, 1881, 14, 2745.**Umbelliferone** (7-Hydroxycoumarin)

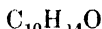
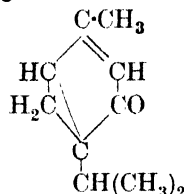
MW, 162

Occurs widely in plants. Needles from  $\text{H}_2\text{O}$ . M.p.  $223-4^\circ$ . Sol. 100 parts boiling  $\text{H}_2\text{O}$ . Very sol. EtOH,  $\text{CHCl}_3$ . Sol. AcOH,  $\text{HCl}$ . Spar. sol.  $\text{Et}_2\text{O}$ . Sol. alkalis and conc.  $\text{H}_2\text{SO}_4$  with blue fluor. Sublimes. Forms Na salt. Reduces warm  $\text{AgNO}_3$ .

*Me ether*: see Herniarin.

*Et ether*:  $\text{C}_{11}\text{H}_{10}\text{O}_3$ . MW, 190. Plates. M.p.  $88^\circ$ . Very sol. EtOH, AcOH,  $\text{C}_6\text{H}_6$ .

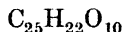
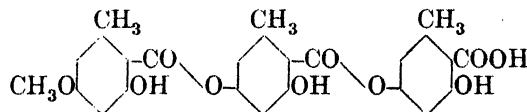
*Acetyl*: prisms or needles from  $\text{H}_2\text{O}$ . M.p.  $140^\circ$ .

Grimaux, *Bull. soc. chim.*, 1895, 13, 900.**Umbellulone**

MW, 150

Constituent of essential oil from *Umbellularia californica*, Meissn. Colourless oil. B.p.  $219-20^\circ/749$  mm.,  $92.5-93^\circ/10$  mm.  $D_{20}^{25} 0.9581$ .  $n_D^{20} 1.48325$ .  $[\alpha]_D - 37^\circ$ .

*Semicarbazone*: cryst. from MeOH. Decomp. at  $240-3^\circ$ .

Power, Lees, *J. Chem. Soc.*, 1904, 85, 636.**Umbilicaric Acid** (Monomethyl ether of gyrophoric acid)

MW, 482

Occurs in numerous lichens. Plates from EtOH. M.p.  $185-6^\circ$ . Sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{Me}_2\text{CO}$ .Aq. Insol.  $\text{H}_2\text{O}$ .

Asahina, Yosioka, *Ber.*, 1937, 70, 204.Koller, Pfeiffer, *Monatsh.*, 1933, 62, 359.**Undecanal.**

See Undecyl Aldehyde.

**Undecane** (Hendecane)

MW, 156

Constituent of natural petroleum. Liq. B.p.  $194.5^\circ$ ,  $81^\circ/15$  mm.,  $61^\circ/2$  mm.  $D_{20}^{20} 0.7411$ .  $n_D^{20} 1.41862$ .

Hess, Bappert, *Ann.*, 1925, 441, 151.Clemmensen, *Ber.*, 1913, 46, 1841.**Undecane-1-carboxylic Acid.**

See Lauric Acid.

**Undecane-1 : 11-dicarboxylic Acid.**

See Brassylic Acid.

**Undecanol-1.**

See Undecyl Alcohol.

**Undecanol-2.**

See Methylnonylcarbinol.

**Undecanol-3.**

See Ethyloctylcarbinol.

**Undecanone-2.**

See Methyl nonyl Ketone.

**Undecanone-3.**

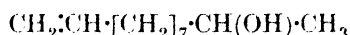
See Ethyl octyl Ketone.

**Undecanone-4.**

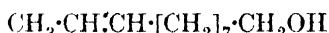
See Propyl heptyl Ketone.

**Undecanone-6.**

See n-Caprone.

**1-Undecenol-10**C<sub>11</sub>H<sub>22</sub>O MW, 170Constituent of essential oil of *Litsea odorifera*,  
Val. B.p. 233°. D<sub>4</sub><sup>20</sup> 0.835.Romburgh, *Chem. Zentr.*, 1911, II, 1863.**1-Undecenol-11.**

See Undecenyl Alcohol.

**2-Undecenol-11 (Isoundecylenic alcohol)**C<sub>11</sub>H<sub>22</sub>O MW, 170Liq. B.p. 248.5°, 124–5°/8 mm. D<sub>4</sub><sup>15</sup> 0.8507.  
n<sub>D</sub><sup>19</sup> 1.4535.Acetyl: b.p. 127–8°/7 mm. D<sub>4</sub><sup>15</sup> 0.8841.Phenylurethane: needles from EtOH. Aq. M.p.  
65–6°.Chuit, Boelsing, Hausser, Malet, *Helv.*  
*Chim. Acta*, 1926, 9, 1085.**Undecenyl Alcohol (1-Undecenol-11, unde-**  
**cylenic alcohol)**C<sub>11</sub>H<sub>22</sub>O MW, 170Liq. solidifying to leaflets on cooling. F.p.  
– 2°. B.p. 250°, 132–3°/15 mm., 122°/3 mm.  
D<sub>4</sub><sup>15</sup> 0.8495. n<sub>D</sub><sup>19</sup> 1.4506.Acetyl: liq. B.p. 125–7°/7 mm. D<sub>4</sub><sup>20</sup> 0.8808.Phenylurethane: needles from EtOH. M.p.  
54.5–55°.

Allophanate: m.p. 143°.

Grün, Wirth, *Ber.*, 1922, 55, 2208.Bouveault, Blanc, *Bull. soc. chim.*, 1904,  
31, 1210.**Undecenylamine (11-Amino-1-undecylene,**  
**11-amino-1-undecene)**C<sub>11</sub>H<sub>23</sub>N MW, 169

Liq. B.p. 238–40°, 123°/16 mm.

N-Benzoyl: plates from C<sub>6</sub>H<sub>6</sub>. M.p. 41–2°.Krafft, Tritschler, *Ber.*, 1900, 33, 3581.**1-Undecine.**

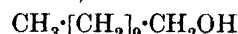
See Nonylacetylene.

**2-Undecine.**

See Methyloctylacetylene.

**Undecoic Acid.**

See Undecylic Acid.

**Undecyl Alcohol (Undecanol-1, hendecyl**  
**alcohol, hendecanol-1)**C<sub>11</sub>H<sub>24</sub>O MW, 172F.p. 19° (11°). B.p. 147°/25 mm., 131°/  
15 mm., 123–5°/6 mm. D<sub>4</sub><sup>23</sup> 0.8334. n<sub>D</sub><sup>23</sup> 1.4392.Phenylurethane: needles from EtOH. M.p.  
62°.

o-Nitrophenylurethane: m.p. 37°.

m-Nitrophenylurethane: plates from EtOH.  
M.p. 56°.

p-Nitrophenylurethane: m.p. 99.5°.

3:5-Dinitrophenylurethane: m.p. 62°.

Allophanate: cryst. from EtOH. M.p. 155.5–  
156°.Ford, Marvel, *Organic Syntheses*, 1930,  
X, 63.Levene, West, Allen, Scheer, *J. Biol.*  
*Chem.*, 1915, 23, 72.Hoeke, *Rec. trav. chim.*, 1935, 54, 505.**Undecyl Aldehyde (Undecylic aldehyde,**  
**undecanal)**C<sub>11</sub>H<sub>22</sub>O MW, 170F.p. – 4°. B.p. 116–17°/18 mm. D<sub>4</sub><sup>23</sup> 0.8251.  
n<sub>D</sub><sup>23</sup> 1.4322. Readily polymerises. Oxidises in  
air to undecylic acid.

Oxime: needles from MeOH. Aq. M.p. 72°.

Semicarbazone: cryst. from MeOH. M.p. 103°.

Azine: cryst. from MeOH. M.p. 57°.

2:4-Dinitrophenylhydrazones: yellow cryst.  
from EtOH. M.p. 104°.Darzens, Levy, *Compt. rend.*, 1933, 196,  
348.Blaise, Guerin, *Bull. soc. chim.*, 1903, 29,  
1202.Blaise, *Bull. soc. chim.*, 1904, 31, 492.**n-Undecylamine (1-Aminoundecane, hende-**  
**cylamine)**C<sub>11</sub>H<sub>25</sub>N MW, 171M.p. 15–16°. B.p. 231–2°/727 mm. Sol. hot  
H<sub>2</sub>O, EtOH. Insol. Et<sub>2</sub>O. Volatile in steam.



*B, HCl*: cryst. from conc. HCl. M.p. 190°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow plates from H<sub>2</sub>O. Decomp. about 180°.

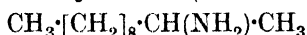
*N-Acetyl*: needles from pet. ether. M.p. 48°. B.p. 192°/12 mm. *B, HCl*: cryst. M.p. 65–6°.

*N-Benzoyl*: needles from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 60°.

Naegeli, Grüntuch, Lendorff, *Helv. Chim. Acta*, 1929, 12, 227.

Jeffreys, *Am. Chem. J.*, 1899, 22, 33.

#### sec.-n-Undecylamine (2-Aminoundecane)



C<sub>11</sub>H<sub>25</sub>N MW, 171

B.p. 231°/741 mm., 113°/26 mm. Absorbs CO<sub>2</sub> readily.

*B, HCl*: needles from ligroin. M.p. 84°.

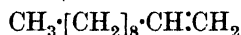
*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow tablets from EtOH. Darkens at about 240°.

*N-Acetyl*: needles from EtOH.Aq. M.p. 58°.

*Picrate*: m.p. 111°.

Thoms, Mannich, *Ber.*, 1903, 36, 2554.

#### 1-Undecylene (Undecene-1, hendecene-1)

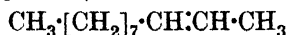


C<sub>11</sub>H<sub>22</sub> MW, 154

B.p. 192–5°, 84°/18 mm. D<sub>20</sub><sup>20</sup> 0.7787. n<sub>D</sub><sup>20</sup> 1.4440.

Ruhemann, *Z. angew. Chem.*, 1931, 44, 78.

#### 2-Undecylene (Undecene-2, hendecene-2)



C<sub>11</sub>H<sub>22</sub> MW, 154

B.p. 192–3°, 78.5°/14 mm. D<sub>15</sub><sup>15</sup> 0.7735. n<sub>D</sub><sup>15</sup> 1.43325.

Thoms, Mannich, *Ber.*, 1903, 36, 2548.

#### 1-Undecylenic Acid



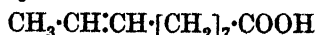
C<sub>11</sub>H<sub>20</sub>O<sub>2</sub> MW, 184

*Nitrile*: C<sub>11</sub>H<sub>19</sub>N. MW, 165. Two stereoisomeric forms. (i) B.p. 119.7–119.9°/10 mm. D<sub>20</sub><sup>20</sup> 0.83255. n<sub>D</sub><sup>20</sup> 1.44816. (ii) B.p. 127.9–128.1°/10 mm. D<sub>20</sub><sup>20</sup> 0.83359. n<sub>D</sub><sup>20</sup> 1.45146.

*Amide*: C<sub>11</sub>H<sub>21</sub>ON. MW, 183. Two stereoisomeric forms corresponding to the above nitriles. (i) Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 76–7°. (ii) Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 114–15°.

Caillie, *Chem. Zentr.*, 1936, I, 4423.

#### 8-Undecylenic Acid



C<sub>11</sub>H<sub>20</sub>O<sub>2</sub> MW, 184

M.p. 19°. B.p. 273–5°, 165°/10 mm. D<sub>0</sub><sup>25</sup> 0.9119. CrO<sub>3</sub> → azelaic acid.

*Cu salt*: m.p. 229–30°.

*Zn salt*: m.p. 111–12°.

*Pb salt*: m.p. 78°.

*Et ester*: C<sub>13</sub>H<sub>24</sub>O<sub>2</sub>. MW, 212. B.p. 258°, 207–8°/185 mm. D<sub>0</sub><sup>20</sup> 0.8966.

*Amide*: C<sub>11</sub>H<sub>21</sub>ON. MW, 183. Cryst. from Et<sub>2</sub>O. M.p. 77–78.5°.

Krafft, Seldis, *Ber.*, 1900, 33, 3572.

#### 9-Undecylenic Acid



C<sub>11</sub>H<sub>20</sub>O<sub>2</sub> MW, 184

Cryst. M.p. 24.5°. B.p. 275°, 165°/15 mm. D<sub>25</sub><sup>25</sup> 0.9102. n<sub>a</sub><sup>24</sup> 1.44642.

*Cu salt*: m.p. 232–4°.

*Pb salt*: m.p. 80°.

*Zn salt*: m.p. 115–16°.

*Me ester*: C<sub>12</sub>H<sub>22</sub>O<sub>2</sub>. MW, 198. M.p. –27.5°. B.p. 248°, 124°/10 mm. D<sub>15</sub><sup>15</sup> 0.889. n<sub>D</sub><sup>20</sup> 1.43928.

*Et ester*: C<sub>13</sub>H<sub>24</sub>O<sub>2</sub>. MW, 212. B.p. 263.5–265.5°, 131.5°/16 mm. D<sub>25</sub><sup>25</sup> 0.87658. n<sub>D</sub><sup>25</sup> 1.4449.

*Anhydride*: C<sub>22</sub>H<sub>38</sub>O<sub>3</sub>. MW, 350. M.p. 13–13.5°.

*Chloride*: C<sub>11</sub>H<sub>19</sub>OCl. MW, 202.5. Liq. B.p. 128.5°/14 mm.

*Amide*: C<sub>11</sub>H<sub>21</sub>ON. MW, 183. Plates from EtOH. M.p. 87°.

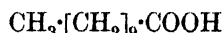
*Nitrile*: C<sub>11</sub>H<sub>19</sub>N. MW, 165. Liq. B.p. 257°, 129–30°/14 mm.

Krafft, Brunner, *Ber.*, 1884, 17, 2985.

#### Undecylenic Alcohol.

See Undecenyl Alcohol.

**Undecylic Acid** (*Undecoic acid, hendecanoic acid*)



C<sub>11</sub>H<sub>22</sub>O<sub>2</sub> MW, 186

Cryst. from Me<sub>2</sub>CO. M.p. 29.5–30.5°. B.p. 212.5°/100 mm., 164°/15 mm. Ag salt insol. H<sub>2</sub>O. Ba salt spar. sol. H<sub>2</sub>O.

*Me ester*: C<sub>12</sub>H<sub>24</sub>O<sub>2</sub>. MW, 200. B.p. 123°/9–10 mm.

*Et ester*: C<sub>13</sub>H<sub>26</sub>O<sub>2</sub>. MW, 214. B.p. 140°/20 mm.

*p-Chlorophenacyl ester*: plates from EtOH. M.p. 60.2°.

*p-Bromophenacyl ester*: plates from EtOH. M.p. 68.2°.

*p-Iodophenacyl ester*: plates from EtOH. M.p. 81.8°.

*Amide*: C<sub>11</sub>H<sub>23</sub>ON. MW, 185. Cryst. from EtOH. M.p. 103°.

*Nitrile*: C<sub>11</sub>H<sub>21</sub>N. MW, 167. Liq. B.p. 253–4°.

*Anilide*: cryst. from EtOH. M.p. 71°.

*o*-Toluidide : m.p. 78°.

*p*-Toluidide : cryst. from EtOH. M.p. 80°.

Moses, Reid, *J. Am. Chem. Soc.*, 1932, **54**, 2101.

Asano, *Chem. Abstracts*, 1922, **16**, 1931.

Robertson, *J. Chem. Soc.*, 1919, **115**, 1210.

Levene, West, *J. Biol. Chem.*, 1914, **18**, 464.

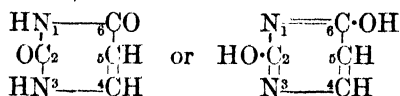
**Undecyl phenyl Ketone.**

See Laurophenone.

**Ungernine.**

See Tazettine.

**Uracil** (2 : 6-Dihydroxypyrimidine, 2 : 6-pyrimidinedione)



$\text{C}_4\text{H}_4\text{O}_2\text{N}_2$  MW, 112

Needles from  $\text{H}_2\text{O}$ . M.p. 335°. Sol. hot  $\text{H}_2\text{O}$ , ammonia. Spar. sol. cold  $\text{H}_2\text{O}$ . Forms K salt.

*N*-Me : see Methyluracil.

*N*-Di-Me : see Dimethyluracil.

1-*N*-Et :  $\text{C}_6\text{H}_8\text{O}_2\text{N}_2$ . MW, 140. Prisms from  $\text{C}_6\text{H}_6$ . M.p. 173-4°. Sol. hot  $\text{H}_2\text{O}$ , EtOH.

1-*N*-Et-3-*N*-Me : see Methylethyluracil.

1 : 3-*N*-Di-Et :  $\text{C}_8\text{H}_{12}\text{O}_2\text{N}_2$ . MW, 168. M.p. 14-15°. B.p. 290-5°, 135°/4 mm.

1-*N*-Benzyl :  $\text{C}_{11}\text{H}_{10}\text{O}_2\text{N}_2$ . MW, 202. Prisms from  $\text{H}_2\text{O}$ . M.p. 175°.

3-*N*-Benzyl : prisms from EtOH. M.p. 173°. Very sol. hot AcOH. Sol.  $\text{Me}_2\text{CO}$ . Spar. sol.  $\text{H}_2\text{O}$ ,  $\text{CHCl}_3$ . Insol.  $\text{Et}_2\text{O}$ , cold  $\text{C}_6\text{H}_6$ , dil. min. acids.

Hilbert, Johnson, *J. Am. Chem. Soc.*, 1930, **52**, 2001.

Davidson, Baudisch, *J. Am. Chem. Soc.*, 1926, **48**, 2382.

Gabriel, *Ber.*, 1905, **38**, 1690.

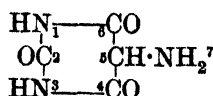
**Uracil-4-carboxylic Acid.**

See Orotic Acid.

**Uradal.**

See Adalin.

**Uramil** (5-Aminobarbituric acid)



$\text{C}_4\text{H}_6\text{O}_3\text{N}_3$  MW, 143

Needles or plates from  $\text{H}_2\text{O}$ . Does not melt below 400°. Mod. sol. hot  $\text{H}_2\text{O}$ . Insol. cold  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ . Sol. conc.  $\text{H}_2\text{SO}_4$ , dil. aq. KOH,  $\text{NH}_3$ . Darkens in air.

Forms Na, K, Ba, Pb salts. Pb salt spar. sol.  $\text{H}_2\text{O}$ .

1 : 3-*N*-Di-Me :  $\text{C}_6\text{H}_9\text{O}_3\text{N}_3$ . MW, 171. Needles. M.p. about 200° decomp. Turns red in air. Spar. sol. cold  $\text{H}_2\text{O}$ . Insol. EtOH. Sol. dil. acids. Reduces  $\text{AgNO}_3$  and Fehling's. Decomp. by alkalis.

1 : 3 : 7-*N*-Tri-Me :  $\text{C}_7\text{H}_{11}\text{O}_3\text{N}_3$ . MW, 185. Needles from  $\text{H}_2\text{O}$  or EtOH. M.p. about 200° decomp. Sol. hot  $\text{H}_2\text{O}$ . Spar. sol. EtOH. Reduces  $\text{AgNO}_3$ .

1 : 3 : 7 : 7-*N*-Tetra-Me :  $\text{C}_8\text{H}_{13}\text{O}_3\text{N}_3$ . MW, 199. Cryst. from EtOH. M.p. 230-2° decomp. Mod. sol.  $\text{H}_2\text{O}$ . Spar. sol. MeOH, EtOH. Very spar. sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Monohydrate : needles from  $\text{H}_2\text{O}$ . M.p. 225° decomp.

7-*N*-Et :  $\text{C}_6\text{H}_9\text{O}_3\text{N}_3$ . MW, 171. Needles from  $\text{H}_2\text{O}$ . M.p. 297° decomp. Sol. hot  $\text{H}_2\text{O}$ .

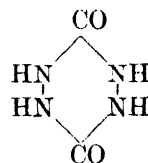
1 : 3-*N*-Di-Et :  $\text{C}_8\text{H}_{13}\text{O}_3\text{N}_3$ . MW, 199. Cryst. M.p. about 200° decomp. Spar. sol.  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{C}_6\text{H}_6$ . Sols decompose on heating.

1 : 3-*N*-Diphenyl :  $\text{C}_{16}\text{H}_{13}\text{O}_3\text{N}_3$ . MW, 295. Needles from EtOH. M.p. 97°. Decomp. in air, becoming red.

7-*N*-Benzyl :  $\text{C}_{11}\text{H}_{11}\text{O}_3\text{N}_3$ . MW, 233. Needles from AcOH. Decomp. about 280°.

Hartmann, Sheppard, *Organic Syntheses*, 1932, XII, 84.

***p*-Urazine** (Dicarbamide, diurea)



$\text{C}_2\text{H}_4\text{O}_2\text{N}_4$  MW, 116

Prisms from  $\text{H}_2\text{O}$ . M.p. about 270° (266°). Spar. sol. EtOH, AcOH, hot  $\text{H}_2\text{O}$ . Reduces  $\text{NH}_3$ ,  $\text{AgNO}_3$ .  $\text{FeCl}_3 \rightarrow$  red col.  $\text{H}_2\text{SO}_4 + \text{HNO}_3 \rightarrow$  violet-red col. Acts as monobasic acid.

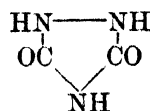
Hydrazine salt : prisms. M.p. 197°.

*N*-Acetyl : plates. Decomp. at 235°.

Purgotti, Viganò, *Gazz. chim. ital.*, 1901, **31**, ii, 550.

Linch, *J. Chem. Soc.*, 1912, **101**, 1756.

**Urazole** (3 : 5-Diketodihydro-1 : 2 : 4-triazole, hydrazodicarbonimide, 3 : 5-diketopyrazolidine)



$\text{C}_2\text{H}_3\text{O}_2\text{N}_3$  MW, 101

Plates from  $\text{H}_2\text{O}$ . M.p.  $244^\circ$  decomp. Very sol.  $\text{H}_2\text{O}$ . Spar. sol.  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ . Reacts acid.

*Acetyl deriv.*: cryst. from  $\text{H}_2\text{O}$ . M.p.  $221.5^\circ$ .

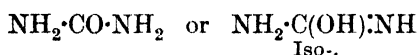
*Diacyl deriv.*: plates from  $\text{EtOH}$ . M.p.  $206^\circ$ .

*Triacyl deriv.*: prisms from  $\text{C}_6\text{H}_6$ . M.p.  $138^\circ$ .

Stollé, Krauch, *J. prakt. Chem.*, 1913, **88**, 314.

Thiele, Stange, *Ann.*, 1894, **283**, 41.

### Urea (*Carbamide, isourea*)



$\text{CH}_4\text{ON}_2$  MW, 60

Constituent of blood and tissue fluids of all vertebrates and of the urine of all mammals. Also occurs in many invertebrates, Nematodes, Crustaceans, Molluscs, etc., and in many fungi and moulds. Prisms with faint salty taste from  $\text{H}_2\text{O}$  or  $\text{EtOH}$ . M.p.  $132^\circ$ . Sol.  $\text{MeOH}$ ,  $\text{EtOH}$ . Insol.  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Sol. 1 part  $\text{H}_2\text{O}$  at  $17^\circ \rightarrow$  neutral sol. Sublimes without decomp. in cathode-ray vacuum. Heat above m.p.  $\rightarrow$  cyanuric acid: at  $150\text{--}70^\circ \rightarrow$  biuret: at  $200^\circ \rightarrow$  triureide of cyanuric acid. Cold aq. alkalis  $\rightarrow$   $\text{NH}_3$ . Hot dil. acids or alkalis  $\rightarrow$   $\text{CO}_2 + \text{NH}_3$ . *p*-Dimethylaminobenzaldehyde +  $\text{HCl}$  (Ehrlich's reagent)  $\rightarrow$  intense yellow col. After heating  $\rightarrow$  biuret reaction with  $\text{NaOH}$  and  $\text{CuSO}_4$ .  $\text{HNO}_2$  or alk.  $\text{NaOBr} \rightarrow$  evolution of  $\text{N}$ .

*B, HCl*: plates. Decomp. at  $145^\circ$ .

*B, HNO<sub>3</sub>*: prisms. M.p.  $152^\circ$  decomp.

*N:N'-Diacyl*: needles from  $\text{EtOH}$ . M.p.  $152\text{--}3^\circ$ .

*N:N'-Dibenzoyl*: needles from  $\text{EtOH}$ . M.p.  $218\text{--}22^\circ$  decomp.

*N-Dibenzoyl*: needles from  $\text{EtOH}$ . M.p.  $197^\circ$ .

*N-Tri-Me*:  $\text{C}_4\text{H}_{10}\text{ON}_2$ . MW, 102. Prisms from  $\text{Et}_2\text{O}$ . M.p.  $75.5^\circ$ . B.p.  $232.5^\circ/764.5$  mm. Very sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ .

*N-Tetra-Me*:  $\text{C}_5\text{H}_{12}\text{ON}_2$ . MW, 116. B.p.  $177.5^\circ/766$  mm. Very sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ .

*N-Tri-Et*:  $\text{C}_7\text{H}_{16}\text{ON}_2$ . MW, 144. Prisms from pet. ether. M.p.  $65^\circ$ . B.p.  $223^\circ$  ( $235^\circ$ ).

*N-Tetra-Et*:  $\text{C}_9\text{H}_{20}\text{ON}_2$ . MW, 172. Liq. with peppermint odour. B.p.  $210\text{--}15^\circ$  ( $205^\circ$ ). Sol. acids, re-ppd. by alkalis. Insol.  $\text{H}_2\text{O}$ .

*N-Triphenyl*:  $\text{C}_{19}\text{H}_{16}\text{ON}_2$ . MW, 288. Plates. M.p.  $136^\circ$ .

*N-Tetraphenyl*:  $\text{C}_{25}\text{H}_{20}\text{ON}_2$ . MW, 364. Cryst. from  $\text{C}_6\text{H}_6$ . M.p.  $183^\circ$ . Sol.  $\text{EtOH}$ .

*O-Me*:  $\text{C}_2\text{H}_6\text{ON}_2$ . MW, 74. M.p.  $44\text{--}5^\circ$ . B.p.  $82^\circ/9$  mm.  $k = 6.4 \times 10^{-5}$  at  $25^\circ$ . Volatile in vapours of  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . *B, HCl*: m.p.  $130^\circ$

decomp. *B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: m.p.  $178^\circ$  decomp. *Picrate*: m.p.  $184^\circ$  decomp. *N-Acetyl*: cryst. from pet. ether. M.p.  $58.5^\circ$ . *N-Benzoyl*: prisms from  $\text{MeOH}$ . Aq. M.p.  $77\text{--}8^\circ$ .

*O-Et*:  $\text{C}_3\text{H}_8\text{ON}_2$ . MW, 88. M.p.  $42^\circ$ . B.p.  $95\text{--}6^\circ/15$  mm. Sol.  $\text{Et}_2\text{O}$ .  $k = 10.4 \times 10^{-5}$  at  $25^\circ$ . *B, HCl*: m.p.  $123\text{--}4^\circ$  decomp. *B<sub>2</sub>, H<sub>2</sub>PtCl<sub>6</sub>*: yellow plates from  $\text{EtOH}$ . M.p.  $178.5^\circ$  decomp. *N-Benzoyl*: cryst. from  $\text{EtOH}$ . Aq. M.p.  $74\text{--}5^\circ$ .

Other urea derivatives are given separately elsewhere.

Foss, *L'Urée* (Paris, 1928).

Werner, *The Chemistry of Urea*, (Monographs on Biochemistry, London, 1923).

Michler, Escherich, *Ber.*, 1879, **12**, 1164.

Wallach, *Ann.*, 1882, **214**, 275.

Schenck, *Z. physiol. Chem.*, 1912, **77**, 383.

Reudler, *Rec. trav. chim.*, 1914, **33**, 64.

### Urea chloride.

See under Carbamic Acid.

### Ureidoacetic Acid.

See Hydantoic Acid.

### 5-Ureidobarbituric Acid.

See  $\psi$ -Uric Acid.

### Ureidoethyl Alcohol.

See *N*-2-Hydroxyethylurea.

### Ureidoformamide.

See Biuret.

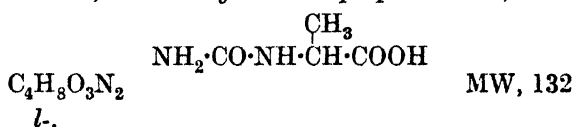
### Ureidoformic Acid.

See Allophanic Acid.

### Ureidophenylacetic Acid.

See 1-Phenylhydantoic Acid.

### 1-Ureidopropionic Acid (*N*-Carbamyl- $\alpha$ -alanine, *N*-carbamyl-1-aminopropionic acid)

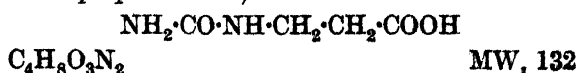


*dl*-. Prisms, needles or plates from  $\text{H}_2\text{O}$ , plates from  $\text{EtOH}$ . M.p.  $185^\circ$  ( $161^\circ$  decomp.). Sol. 100 parts  $\text{EtOH}$  at  $20^\circ$  and 46 parts  $\text{H}_2\text{O}$  at  $20^\circ$ . Insol.  $\text{Et}_2\text{O}$ . Heat at  $140^\circ$  or with dil.  $\text{HCl} \rightarrow$  5-methylhydantoin.

*K salt*: needles +  $1\text{H}_2\text{O}$ . Decomp. at  $200\text{--}5^\circ$ .

Dakin, *J. Chem. Soc.*, 1915, **107**, 439.

### 2-Ureidopropionic Acid (*N*-Carbamyl-2-aminopropionic acid)



Cryst. M.p. 170–1° decomp. Sol. H<sub>2</sub>O. Spar. sol. most org. solvents. Heat at 160–70° or with dil. HCl → 2 : 4-diketohexahydropyrimidine.

*K salt*: cryst. Sinters at 80°, m.p. 100°.

Lengfeld, Stieglitz, *Am. Chem. J.*, 1893, 15, 516.

**Urethane** (*Ethyl carbamate, aminoformic acid ethyl ester*)



C<sub>3</sub>H<sub>7</sub>O<sub>2</sub>N

MW, 89

Prisms from C<sub>6</sub>H<sub>6</sub> or toluene. M.p. 49–50°. B.p. 184°. Very sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Spar. sol. ligroin. Heat aq. sol. to 130° → urea. Heat with NaOH → NaCNO + Na<sub>2</sub>CO<sub>3</sub> + C<sub>2</sub>H<sub>5</sub>OH.

*N-Nitroso*: C<sub>3</sub>H<sub>6</sub>O<sub>3</sub>N<sub>2</sub>. MW, 118. Yellow needles from ligroin. M.p. 51–2° decomp. Very sol. MeOH, EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO. Sol. H<sub>2</sub>O. Spar. sol. ligroin.

*N-Chloroacetyl*: cryst. from EtOH. M.p. 129°.

*N-Bromoacetyl*: cryst. from EtOH. M.p. 119°.

*N-Dichloroacetyl*: needles from H<sub>2</sub>O. M.p. 98°.

*N-p-Nitrobenzoyl*: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 152°.

Basterfield, Greig, *Chem. Zentr.*, 1933, II, 1021.

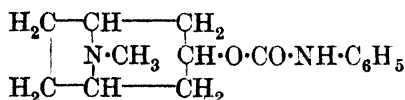
Basterfield, Woods, Wright, *J. Am. Chem. Soc.*, 1926, 48, 2371.

Guerci, *Chem. Abstracts*, 1922, 16, 2481.

**Urethylan.**

Methyl carbamate. See under Carbamic Acid.

**Uretropine** (*Phenylcarbamic tropine ester, anilinoformyltropine*)



C<sub>15</sub>H<sub>20</sub>O<sub>2</sub>N<sub>2</sub>

MW, 260.

Prisms from Et<sub>2</sub>O. M.p. 171–2°. Very sol. EtOH, Et<sub>2</sub>O. Spar. sol. cold C<sub>6</sub>H<sub>6</sub>. Has mydriatic action.

*B,HCl*: prisms from EtOH.Aq. M.p. 289–90°.

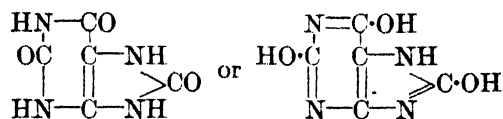
*B<sub>2</sub>,H<sub>2</sub>SO<sub>4</sub>*: needles + 4H<sub>2</sub>O from H<sub>2</sub>O. M.p. 201° decomp.

*B,H AuCl<sub>4</sub>*: orange-red cryst. powder. M.p. 188–9°.

*Picrate*: yellow prisms from EtOH.Aq. M.p. 223–4°.

Jowett, Pyman, *J. Chem. Soc.*, 1909, 95, 1027.

**Uric Acid** (2 : 6 : 8-Trihydroxypurine)



C<sub>5</sub>H<sub>4</sub>O<sub>3</sub>N<sub>4</sub>

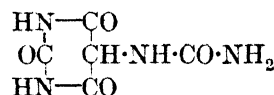
MW, 168

Chief end-product of purine metabolism in man. Constituent of urine of animals, excrement of birds, reptiles, insects, etc. Rhombic prisms or plates. Insol. EtOH, Et<sub>2</sub>O. Very spar. sol. H<sub>2</sub>O. Sol. glycerol. Spar. sol. min. acids. Readily sol. alkalis. Heat → urea, cyanuric acid, HCN and NH<sub>3</sub>. KOH fusion → KCN, KCNO, K<sub>2</sub>CO<sub>3</sub> and (COOK)<sub>2</sub>. HNO<sub>3</sub> → alloxan + urea. KMnO<sub>4</sub> → allantoin. Acts as weak dibasic acid. In alk. sol. reduces Ag and Cu salts, phosphomolybdates and phosphotungstates. Gives murexide reaction.

Traube, *Ber.*, 1900, 33, 1371, 3035.

Fischer, *Ber.*, 1897, 30, 559.

**ψ-Uric Acid** (5-Ureidobarbituric acid)



C<sub>5</sub>H<sub>6</sub>O<sub>4</sub>N<sub>4</sub>

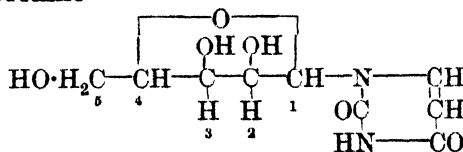
MW, 186

Prisms. Spar. sol. H<sub>2</sub>O. Sol. aq. alkalis. Heat of comb. C, 455.2 Cal. HNO<sub>3</sub> → alloxan + urea. Alk. KMnO<sub>4</sub> → alloxanic acid. Heat with oxalic acid or with hot dil. HCl → uric acid.

Biltz, *Ann.*, 1921, 423, 119.

Fischer, *Ber.*, 1897, 30, 570, 3091.

**Uridine**



C<sub>9</sub>H<sub>12</sub>O<sub>6</sub>N<sub>2</sub>

MW, 244

Needles from EtOH.Aq. M.p. 165°. [α]<sub>D</sub><sup>20</sup> + 4.0° in H<sub>2</sub>O.

*5-Phosphate*: prisms from MeOH. M.p. 198–5°. [α]<sub>D</sub><sup>20</sup> + 9.5° in H<sub>2</sub>O.

*N-Me*: C<sub>10</sub>H<sub>14</sub>O<sub>6</sub>N<sub>2</sub>. MW, 258. Plates. M.p. 108–10°. Sol. MeOH, EtOH, Me<sub>2</sub>CO, AcOH, H<sub>2</sub>O, Py. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>.

*2 : 3-Di-Me ether*: C<sub>11</sub>H<sub>16</sub>O<sub>6</sub>N<sub>2</sub>. MW, 272.

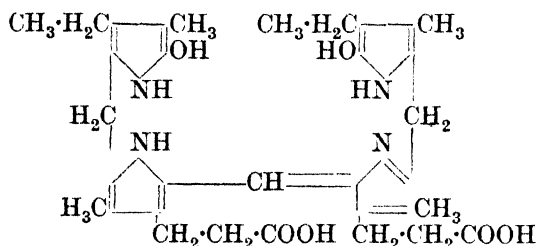
Cryst. from AcOEt. M.p. 168–9°. Sol. H<sub>2</sub>O, EtOH, MeOH, AcOH, Me<sub>2</sub>CO, Py.  $[\alpha]_D^{25} + 68.0^\circ$  in Me<sub>2</sub>CO.

2:3-Di-p-toluenesulphonyl: needles from EtOH. M.p. 199°.  $[\alpha]_D^{25} - 26.6^\circ$  in Me<sub>2</sub>CO.

Levene, Tipson, *J. Biol. Chem.*, 1934, **104**, 385; **105**, 419.

Levene, Jacobs, *Ber.*, 1910, **43**, 3158.

### Urobilin (Stereobilin)



C<sub>33</sub>H<sub>24</sub>O<sub>6</sub>N<sub>4</sub>

MW, 572

Also known as Urobilin IX- $\alpha$ . Constituent of urine and faeces. Yellow needles from CHCl<sub>3</sub> or MeCO. M.p. 177°. Very sol. MeOH, EtOH, AcOH, Py. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, AcOEt, C<sub>6</sub>H<sub>6</sub>. Insol. CCl<sub>4</sub>, pet. ether. Very sol. amyl alcohol with green fluor. Very sol. conc. HCl and conc. H<sub>2</sub>SO<sub>4</sub>  $\rightarrow$  red sols.

B,HCl: prisms from Me<sub>2</sub>CO, m.p. 199–200°; prisms from CHCl<sub>3</sub>, m.p. 147–71°.

B,HBr: prisms from Me<sub>2</sub>CO. M.p. 200–1°.

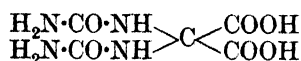
Siedel, Meier, *Z. physiol. Chem.*, 1936, **242**, 101.

Heilmeyer, Krebs, *Z. physiol. Chem.*, 1934, **228**, 33, 46.

### Urotropine.

See Hexamethylenetetramine.

### Uroxic Acid (Diureidomalonic acid)



C<sub>5</sub>H<sub>8</sub>O<sub>6</sub>N<sub>4</sub>

MW, 220

Needles from C<sub>6</sub>H<sub>6</sub>. Decomp. at 162°. Sol. H<sub>2</sub>O, EtOH, Et<sub>2</sub>O. Spar. sol. C<sub>6</sub>H<sub>6</sub>. Insol. pet. ether. Loses CO<sub>2</sub> in H<sub>2</sub>O at 60°. Ag salt spar. sol. H<sub>2</sub>O.

NH<sub>4</sub> salt: prisms. Decomp. at 182°.

Di-Et ester: C<sub>9</sub>H<sub>16</sub>O<sub>8</sub>N<sub>4</sub>. MW, 276. Cryst. from EtOH. M.p. 170°.

Biltz, Robl, *Ber.*, 1920, **53**, 1950.

### Ursol P.

See p-Aminophenol.

### Ursolic Acid (Ursone, prunol, malol, malolic acid)

C<sub>30</sub>H<sub>48</sub>O<sub>3</sub>

MW, 456

Constituent of leaves and skins of apples and pears, leaves of *Rhododendron hymenanthes*, leaves of *Epigaea asiatica*, Maxim, etc. Prisms from Et<sub>2</sub>O. M.p. 285° (291°). Sol. hot AcOH, EtOH. Mod. sol. Et<sub>2</sub>O, AcOH, Me<sub>2</sub>CO, AcOEt, CHCl<sub>3</sub>. Insol. H<sub>2</sub>O, pet. ether. Liebermann-Salkowski reagent  $\rightarrow$  blue col.

Me ester: C<sub>31</sub>H<sub>50</sub>O<sub>3</sub>. MW, 470. Two forms. (i) Cryst. from EtOH.Aq. M.p. 170.5–171.5° (173°). Sol. cold Me<sub>2</sub>CO. (ii) M.p. 230°. Insol. cold Me<sub>2</sub>CO. Benzoyl: m.p. 215–16°. Acetyl: m.p. 246–7°.

Phenacyl ester: m.p. 199–200°.

Drake, Duvall, *J. Am. Chem. Soc.*, 1936, **58**, 1687.

Fujii, Shimada, *J. Pharm. Soc. Japan*, 1935, **55**, 106.

Kuwada, Matsumoto, *Chem. Abstracts*, 1933, **27**, 3925.

Sando, *J. Biol. Chem.*, 1931, **90**, 477; 1923, **56**, 457.

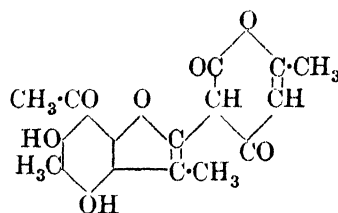
Haar, *Rec. trav. chim.*, 1924, **43**, 542.

Kuwada, Matsukawa, *Chem. Abstracts*, 1934, **28**, 4739.

### Ursone.

See Ursolic acid.

### Usnic Acid



C<sub>18</sub>H<sub>16</sub>O<sub>7</sub>

MW, 344

d.

Occurs in various species of lichen. Constituent of Chinese drug "Shi-koa." Cryst. from MeOH or C<sub>6</sub>H<sub>6</sub>. M.p. 202–4° (195–6°). Very sol. CHCl<sub>3</sub>. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.  $[\alpha]_D^{25} + 508.3^\circ$ .

Diacetyl: needles from MeOH. M.p. 199–200°.  $[\alpha]_D^{25} + 205^\circ$ . FeCl<sub>3</sub>  $\rightarrow$  red col.

l.

M.p. 195–7°. Very sol. CHCl<sub>3</sub>. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.  $[\alpha]_D^{20} - 495^\circ$ .

dl-.

M.p. 193-4°.  $\text{FeCl}_3 \longrightarrow$  brown col.

Diacetyl: m.p. 199-200°.

Curd, Robertson, *J. Chem. Soc.*, 1937, 894.Asahina, Yanagita, *Ber.*, 1937, 70, 66; 1936, 69, 1646.Shöpf, Kraus, Heuck, *Ann.*, 1927, 459, 263.Nakao, *Chem. Abstracts*, 1923, 17, 3184.**Uvaleral.**

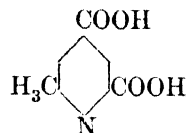
See 1-Bromoisovalerylurea.

**Uvinic Acid.**

See Pyrotartaric Acid.

**Uvitic Acid.**

See 5-Methylisophthalic Acid.

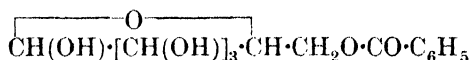
**Uvitonic Acid** ( $\alpha$ -Picoline-4 : 6-dicarboxylic acid, 6-methyl-lutidinic acid) $\text{C}_8\text{H}_7\text{O}_4\text{N}$ 

MW, 181

Cryst. from  $\text{H}_2\text{O}$ . M.p. 282° decomp. Sol. hot  $\text{AcOH}$ , phenol, aniline. Sol. conc.  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{NH}_4\text{OH}$ . Spar. sol. hot  $\text{CHCl}_3$ , amyl alcohol. Very spar. sol. cold  $\text{H}_2\text{O}$ . Insol.  $\text{CS}_2$ ,  $\text{C}_6\text{H}_6$ .

Böttinger, *Ber.*, 1880, 13, 2032.

## V

**Vacciniin** (6-Benzoyl-d-glucose) $\text{C}_{13}\text{H}_{16}\text{O}_7$ 

MW, 284

Amorph. hygroscopic mass with bitter taste. Cryst. from  $\text{Me}_2\text{CO}$  as monohydrate. M.p. 104-6°. Sol.  $\text{H}_2\text{O}$ ,  $\text{MeOH}$ ,  $\text{EtOH}$ ,  $\text{AcOEt}$ ,  $\text{Me}_2\text{CO}$ ,  $\text{Py}$ . Spar. sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Insol.  $\text{Et}_2\text{O}$ , pet. ether.  $[\alpha]_D^{21} + 48^\circ$  in  $\text{EtOH}$ ,  $[\alpha]_D^{21}$  (hydrate) + 45.76° in  $\text{H}_2\text{O}$  (after 10 minutes). Reduces Fehling's.

*Phenylhydrazone*: yellow cryst. from  $\text{EtOH.Aq.}$  M.p. 146-7° (136°).

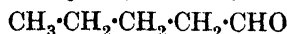
*Phenylosazone*: yellow needles from  $\text{EtOH.Aq.}$  M.p. 141°.

1 : 2 : 3 : 4-*Tetra-acetyl*: cryst. from  $\text{EtOH.}$  M.p. 136°.

Brigl, Zerrweck, *Z. physiol. Chem.*, 1934, 229, 117.

Ohle, *Biochem. Z.*, 1922, 131, 611.

Fischer, Noth, *Ber.*, 1918, 51, 326.

**n-Valeraldehyde** (Pentanal, valeric aldehyde) $\text{C}_5\text{H}_{10}\text{O}$ 

MW, 86

Liq. B.p. 102.5-103.0°.  $D_4^{20}$  0.80952.  $n_D^{20}$  1.39436.

*Di-Et acetal*:  $\text{C}_9\text{H}_{20}\text{O}_2$ . MW, 160. B.p. 59°/12 mm.  $D_4^{22}$  0.829.  $n_D^{22}$  1.4029.

*Oxime*: m.p. 52°.

*Thiosemicarbazone*: cryst. from  $\text{EtOH.Aq.}$ , or  $\text{Et}_2\text{O}$ . M.p. 65°.

*Phenylsemicarbazone*: m.p. 126-7°.

2 : 4-Dinitrophenylhydrazone: yellow cryst. M.p. 98°.

*o-Tolylsemicarbazone*: m.p. 131-2°.

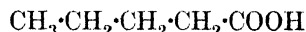
*p-Tolylsemicarbazone*: m.p. 157-8°.

Lieben, Rossi, *Ann.*, 1871, 159, 70.

Bruylants, Ernould, *Chem. Abstracts*, 1932, 26, 3232.

Blaise, *Compt. rend.*, 1904, 138, 698.

**n-Valeric Acid** (Valereanic acid, butane-1-carboxylic acid, propylacetic acid)

 $\text{C}_5\text{H}_{10}\text{O}_2$ 

MW, 102

Liq. with unpleasant odour. M.p. -34.5°. B.p. 186-35°, 96°/23 mm., 86-8°/15 mm. Sol. 27 parts  $\text{H}_2\text{O}$  at 16°.  $D_4^{20}$  0.9387.  $n_D^{19}$  1.4070. Heat of comb. 681.8 Cal.  $k = 1.56 \times 10^{-5}$  at 25°.

*Me ester*:  $\text{C}_6\text{H}_{12}\text{O}_2$ . MW, 116. B.p. 127.3°.  $D_4^0$  0.9097.

*Et ester*:  $\text{C}_7\text{H}_{14}\text{O}_2$ . MW, 130. B.p. 144.6°/736.5 mm.  $D_4^{20}$  0.8765.

*Propyl ester*:  $\text{C}_8\text{H}_{16}\text{O}_2$ . MW, 144. B.p. 167.5°.  $D_4^0$  0.8888.

*n-Butyl ester*:  $\text{C}_9\text{H}_{18}\text{O}_2$ . MW, 158. B.p. 185.8°.  $D_4^0$  0.8847.

*d-sec-Butyl ester*: B.p. 67°/18 mm.  $D_4^{13}$  0.8650.  $n_D^{20}$  1.4070.  $[\alpha]_D^{20} + 20.72^\circ$ .

*n-Amyl ester*:  $\text{C}_{10}\text{H}_{20}\text{O}_2$ . MW, 172. B.p. 204°.  $D_4^0$  0.881.

*Isoamyl ester*: b.p. 187-90°.

*d-sec-n-Amyl ester*: b.p. 86°/16 mm.  $D_4^{17}$  0.8631.  $n_D^{20}$  1.4115.  $[\alpha]_D^{20} + 16.01^\circ$ .

1-Menthyl ester:  $C_{15}H_{28}O_2$ . MW, 240. B.p.  $141^\circ/15$  mm.  $D_4^{20}$  0.9074.  $[\alpha]_D^{20} - 65.55^\circ$ .

Chloride:  $C_5H_9OCl$ . MW, 120.5. B.p.  $107-110^\circ/756$  mm.  $D_4^{15}$  1.0155.

Bromide:  $C_5H_9OBr$ . MW, 165. B.p.  $64^\circ/66$  mm.

Amide: *n*-valeramide.  $C_5H_{11}ON$ . MW, 101. Plates from EtOH. M.p.  $106^\circ$ . Very sol.  $H_2O$ , EtOH,  $Et_2O$ .

Imide:  $C_{10}H_{19}O_2N$ . MW, 185. Cryst. M.p.  $100^\circ$ . Very sol.  $Et_2O$ . Mod. sol. hot EtOH.

Anhydride:  $C_{10}H_{18}O_3$ . MW, 186. B.p.  $218^\circ/754$  mm.,  $110-111^\circ/15$  mm.  $D_4^{17}$  0.9223.

Nitrile: *n*-valeronitrile.  $C_5H_9N$ . MW, 83. B.p.  $141.1-141.2^\circ/764.7$  mm.  $D_4^{15}$  0.80348.  $n_D^{15}$  1.39913.

Anilide: *n*-valeranilide. Prisms from EtOH, cryst. from pet. ether. M.p.  $63^\circ$ .

*p*-Bromoanilide: m.p.  $108^\circ$ .

*p*-Toluidide: cryst. from EtOH. Plates from pet. ether. M.p.  $74^\circ$ .

1-Naphthalide: cryst. from EtOH. M.p.  $112^\circ$ .

Ivanoff, *Bull. soc. chim.*, 1925, **37**, 293.

Gilman, Kirby, *Organic Syntheses*, Collective Vol. I, 355.

Wolff, *Ann.*, 1881, **208**, 110.

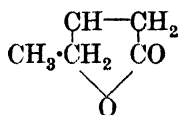
### Valerolactam.

See  $\alpha$ -Piperidone.

### Valerolactinic Acid.

See 1-Hydroxy-*n*-valeric Acid.

$\gamma$ -Valerolactone (3-Methylbutyrolactone, 3-valerolactone)



$C_5H_8O_2$

*dl.*

MW, 100

Constituent of crude pyroligneous acid. F.p.  $-31^\circ$  to leaflets. B.p.  $205-7^\circ$ ,  $102-3^\circ/28$  mm.,  $83-4^\circ/13$  mm.,  $78^\circ/4$  mm. Misc. in all proportions with  $H_2O$ .  $D_4^{25}$  1.0465.  $n_D^{25}$  1.4303.  $HI(+P) \rightarrow n$ -valeric acid.  $HCl \rightarrow 3$ -chloro-*n*-valeric acid. Hyd. by  $Ba(OH)_2$ , slowly by  $H_2O$ .  $NH_3 \rightarrow 3$ -hydroxy-*n*-valeramide.

*d.*

B.p.  $86-90^\circ/14$  mm.  $[\alpha]_D^{20} + 13.5^\circ$ .

Pummerer, Guyot, Birkofer, *Ber.*, 1935, **68**, 490.

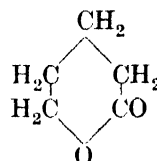
Schuetter, Thomas, *J. Am. Chem. Soc.*, 1930, **52**, 3011.

Levene, Haller, Walti, *J. Biol. Chem.*, 1927, **72**, 591.

Losanitsch, *Monatsh.*, 1914, **35**, 302, 311.

Boorman, Linstead, *J. Chem. Soc.*, 1933, 578.

$\delta$ -Valerolactone (4-Valerolactone, tetrahydro- $\alpha$ -pyrone, tetrahydrocoumalin)



$C_5H_8O_2$

MW, 100

Oil. Solidifies on cooling. M.p.  $-12.5^\circ$ . B.p.  $218-20^\circ$ ,  $113-14^\circ/13-14$  mm.,  $88^\circ/4$  mm. Misc. with EtOH,  $Et_2O$ . Spar. misc. with  $H_2O$ .  $D_4^{20}$  1.0794.  $n_D^{20}$  1.4503. Readily polymerises on standing to several high MW polymers.  $Ba(OH)_2 \rightarrow$  Ba salt of 4-hydroxy-*n*-valeric acid.  $HBr \rightarrow$  4-bromo-*n*-valeric acid.

Coffmann, *J. Am. Chem. Soc.*, 1935, **57**, 1984.

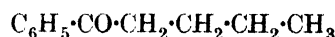
Linstead, Rydon, *J. Chem. Soc.*, 1933, 583.

Fichter, Beisswenger, *Ber.*, 1903, **36**, 1200.

### Valerone.

See Di-isobutyl Ketone.

### Valerophenone (n-Butyl phenyl ketone)



$C_{11}H_{14}O$

MW, 162

Liq. B.p.  $235-42^\circ$ ,  $135-40^\circ/25$  mm.  $D_4^{20}$  0.988.  $n_D^{20}$  1.532.

Oxime: needles from EtOH.Aq. or pet. ether. M.p.  $52.0-52.5^\circ$ . B.p.  $163-5^\circ/13$  mm. Sol. EtOH,  $C_6H_6$ . Spar. sol. pet. ether.

Semicarbazone: needles from EtOH. M.p.  $166^\circ$  ( $157-157.5^\circ$ ).

Shriner, Turner, *J. Am. Chem. Soc.*, 1930, **52**, 1269.

Pfeiffer, Oberlin, *J. prakt. Chem.*, 1924, **108**, 347.

### *n*-Valerylacetic Acid.

See 2-Keto-*n*-heptylic Acid.

### Valerylalcohol.

See under *p*-Hydroxyvalerophenone.

### Valerylcarbinol.

See 1-Hexanolone-2.

### Valerylphenetole.

See under *p*-Hydroxyvalerophenone.

### *p*-Valerylphenol.

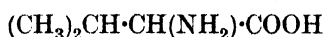
See *p*-Hydroxyvalerophenone.

### 2-*n*-Valerylpropionic Acid.

See 3-Keto-*n*-caprylic Acid.

### Validol.

See under Menthol.

**Valine** (1-Aminoisovaleric acid) $\text{C}_5\text{H}_{11}\text{O}_2\text{N}$ 

MW, 117

d.-.

Plates from EtOH.Aq. M.p. 156–157.5° (293° decomp., sealed tube). Sol. 18.4 parts  $\text{H}_2\text{O}$  at 20°.  $[\alpha]_D^{20} - 29.04^\circ$  in 20% HCl,  $[\alpha]_D^{20} - 39.1^\circ$  in  $\text{Me}_2\text{CO}$ .

Et ester:  $\text{C}_7\text{H}_{15}\text{O}_2\text{N}$ . MW, 145. N-p-Nitrobenzoyl: cryst. from ligroin. M.p. 88°.  $[\alpha]_D^{20} - 3.5^\circ$  in EtOH.

N-Formyl: prisms from hot  $\text{H}_2\text{O}$ . Sinters at 150°, m.p. 156°.  $[\alpha]_D^{20} - 13.07^\circ$  in EtOH, + 16.9° in  $\text{H}_2\text{O}$ .

l.-.

Occurs in pancreas of oxen, in yellow boletus, in seeds of *Lupinus luteus* L., *Vicia sativa* L., *Lupinus angustifolius* L., etc. Plates from EtOH.Aq. M.p. 93–6° (315°, sealed tube). Sol.  $\text{H}_2\text{O}$ . Spar. sol. EtOH.  $[\alpha]_D^{20} + 6.42^\circ$  in  $\text{H}_2\text{O}$ ,  $[\alpha]_{5461}^{21} + 32.77^\circ$  in HCl. Ba(OH)<sub>2</sub>.Aq. at 180° → dl-form.

Et ester: b.p. 63.5°/8 mm. N-Acetyl: viscous liq. B.p. 158°/21 mm. Cryst. in plates on standing.  $D_4^{18} 1.028$ .  $n_D^{18} 1.4517$ .  $[\alpha]_{5461}^{18} - 20.1^\circ$ . N-Benzoyl: needles from pet. ether. M.p. 82°.  $[\alpha]_D^{20} - 3.44^\circ$  in EtOH. N-p-Nitrobenzoyl: needles from pet. ether. M.p. 88°.  $[\alpha]_D^{20} + 4.12^\circ$  in EtOH. N-Benzenesulphonyl: cryst. from pet. ether. M.p. 56°.  $[\alpha]_D^{20} - 1.04^\circ$  in EtOH. N-p-Toluenesulphonyl: cryst. from pet. ether. M.p. 59°.  $[\alpha]_D^{20} + 3.99^\circ$  in EtOH.

N-Formyl: prisms from hot  $\text{H}_2\text{O}$ . Sinters at 150°, m.p. 156°.  $[\alpha]_D^{20} + 13.07^\circ$  in EtOH.

N-Benzoyl: needles from  $\text{H}_2\text{O}$ . M.p. 127°.  $[\alpha]_D^{20} + 17.18^\circ$ .

N-Benzenesulphonyl: needles from EtOH.Aq. M.p. 153°.  $[\alpha]_D^{20} + 19.5^\circ$  in EtOH.

N-p-Toluenesulphonyl: needles from EtOH.Aq. M.p. 147°.  $[\alpha]_D^{20} + 25.0^\circ$  in EtOH.

Picrolonate: m.p. 170–80°.

dl.-.

Plates from EtOH. Sublimes. M.p. about 298° decomp., (sealed tube). Sol. 11.7 parts  $\text{H}_2\text{O}$  at 15°. Insol. cold EtOH, Et<sub>2</sub>O. Cu and Ag salts spar. sol.  $\text{H}_2\text{O}$ .

B,HCl: cryst. from hot  $\text{H}_2\text{O}$ , leaflets from dil. HCl. M.p. 189°.

Et ester: b.p. 174° part. decomp., 82.5°/23 mm., 63.5°/8 mm.  $D_4^{18} 0.9617$ . Very sol.  $\text{H}_2\text{O}$ . B,HCl: hygroscopic cryst. M.p. 76°. N-Acetyl: b.p. 99°/2 mm. Picrate: cryst. M.p. 139.5°.

Butyl ester:  $\text{C}_9\text{H}_{19}\text{O}_2\text{N}$ . MW, 173. Syrup. B.p. 98–98.5°/17 mm.  $D_4^{14} 0.9266$ . Sol.  $\text{H}_2\text{O}$ ,

EtOH, Et<sub>2</sub>O,  $\text{CHCl}_3$ . B,HCl: needles from AcOEt. M.p. 59–60°. Picrate: yellow needles. M.p. 91–2°.

Amide:  $\text{C}_5\text{H}_{12}\text{ON}_2$ . MW, 116. Prisms from  $\text{C}_6\text{H}_6$ . M.p. 78–80°. Very sol.  $\text{H}_2\text{O}$ , EtOH, AcOEt, hot  $\text{C}_6\text{H}_6$ . Spar. sol. Et<sub>2</sub>O, ligroin. Alk. Cu sol. → violet col. B,HBr: cryst. Decomp. at 200°.

Anhydride:  $\text{C}_{10}\text{H}_{20}\text{O}_3\text{N}_2$ . MW, 216. Does not melt below 300°.

Nitrile:  $\text{C}_5\text{H}_{10}\text{N}_2$ . MW, 98. Unstable yellow oil. Very sol.  $\text{H}_2\text{O}$ , EtOH, Et<sub>2</sub>O.

N-Me:  $\text{C}_6\text{H}_{13}\text{O}_2\text{N}$ . MW, 131. Powder. Very sol.  $\text{H}_2\text{O}$ . Spar. sol. hot EtOH. Insol. Et<sub>2</sub>O. Sublimes.

N-Formyl: plates from  $\text{H}_2\text{O}$ . Sinters at 137°, m.p. 140–5°.

N-Benzoyl: m.p. 132.5°.

N-p-Toluenesulphonyl: m.p. 110–11°.

Barrow, Ferguson, *J. Chem. Soc.*, 1935, 415.

Holmes, Adams, *J. Am. Chem. Soc.*, 1934, 56, 2093.

Karrer, Sluys Veer, *Helv. Chim. Acta*, 1932, 15, 746.

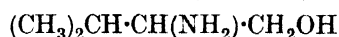
Levene, Bass, Rothen, Steiger, *J. Biol. Chem.*, 1929, 81, 687.

Abderhalden, Landau, *Z. physiol. Chem.*, 1911, 71, 458.

Fischer, Scheibler, *Ber.*, 1908, 41, 2891.

Fischer, *Ber.*, 1906, 39, 2322.

Slimmer, *Ber.*, 1902, 35, 401.

**Valinol** (2-Aminoisoamyl alcohol) $\text{C}_5\text{H}_{13}\text{ON}$ 

MW, 103

d.-.

B,HCl: plates from  $\text{Me}_2\text{CO}$ . M.p. 113°.  $[\alpha]_{5461}^{20} - 16.5^\circ$  in  $\text{H}_2\text{O}$ .

Dibenzoyl deriv.: needles from EtOH.Aq. M.p. 117°.  $[\alpha]_{5461}^{21} + 20.2^\circ$  in Py.

Tribenzoyl: needles. M.p. 119°.  $[\alpha]_{5461}^{20} - 133.8^\circ$  in Py.

l.-.

B,HCl: plates from  $\text{Me}_2\text{CO}$ . M.p. 112–14°.  $[\alpha]_{5461}^{20} + 16.4^\circ$  in  $\text{H}_2\text{O}$ .

Dibenzoyl deriv.: needles. M.p. 117°.  $[\alpha]_{5461}^{20} - 20.1^\circ$  in Py.

Tribenzoyl: needles. M.p. 119°.  $[\alpha]_{5461}^{19} + 133.1^\circ$  in Py.

dl.-.

Oil. B.p. 181–6°/720 mm. Sol.  $\text{H}_2\text{O}$ , EtOH. Mod. sol. Et<sub>2</sub>O. Slowly volatile in steam.

B,HCl: plates from  $\text{Me}_2\text{CO}$ . M.p. 118–19°.



*N-Benzoyl*: needles from pet. ether. M.p. 81–2°.

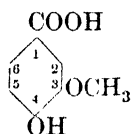
*Dibenzoyl deriv.*: prisms from  $\text{Me}_2\text{CO}$ . Aq. M.p. 114°.

*Tribenzoyl*: plates from EtOH. Aq. M.p. 135°.

Barrow, Ferguson, *J. Chem. Soc.*, 1935, 410.

Karrer *et al.*, *Helv. Chim. Acta*, 1922, 5, 478.

**Vanillic Acid** (*Protocatechuic acid 3-methyl ether, 4-hydroxy-3-methoxybenzoic acid*)



$\text{C}_8\text{H}_8\text{O}_4$

MW, 168

Needles from  $\text{H}_2\text{O}$ . M.p. 210°. Sublimes. Sol. 850 parts  $\text{H}_2\text{O}$  at 14°, 62 parts at 75°, 40 parts at 95°. Sol.  $\text{Et}_2\text{O}$ . Very sol. EtOH.  $k = 3.0 \times 10^{-5}$  at 25°. Salts all very sol.  $\text{H}_2\text{O}$ . No col. with  $\text{FeCl}_3$ . HCl at 150° or KOH fusion  $\rightarrow$  protocatechuic acid. Dist. Ca salt + Ca formate  $\rightarrow$  guaiacol. Loses  $\text{CO}_2$  with aniline at 240°.

*Me ester*:  $\text{C}_9\text{H}_{10}\text{O}_4$ . MW, 182. Needles from EtOH. Aq. M.p. 62–3°. B.p. 285–7°. *Benzoyl*: needles from MeOH. M.p. 104°.

*Et ester*:  $\text{C}_{10}\text{H}_{12}\text{O}_4$ . MW, 196. Needles. M.p. 44°. B.p. 291–3°.

*Propyl ester*:  $\text{C}_{11}\text{H}_{14}\text{O}_4$ . MW, 210. M.p. 43°.

*Isopropyl ester*: m.p. 113.5°.

*Butyl ester*:  $\text{C}_{12}\text{H}_{16}\text{O}_4$ . MW, 224. M.p. 48–9°.

*p-Nitrobenzyl ester*:  $\text{C}_{15}\text{H}_{13}\text{O}_6\text{N}$ . MW, 303. Cryst. from EtOH. Aq. M.p. 140–1° decomp.

*Chloride*:  $\text{C}_8\text{H}_7\text{O}_3\text{Cl}$ . MW, 186.5. *Benzoyl*: needles from pet. ether. M.p. 96–8°.

*Nitrile*:  $\text{C}_8\text{H}_7\text{O}_2\text{N}$ . MW, 149. Needles from  $\text{H}_2\text{O}$ . M.p. 89–90°. Very sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . *Acetyl*: needles from  $\text{H}_2\text{O}$ . M.p. 110°.

*Me ether*: see Veratric Acid.

*Et ether*: 3-methoxy-4-ethoxybenzoic acid.  $\text{C}_{10}\text{H}_{12}\text{O}_4$ . MW, 196. Needles from  $\text{H}_2\text{O}$  or  $\text{C}_6\text{H}_6$ . M.p. 195–6°. Sublimes. Very sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. hot  $\text{H}_2\text{O}$ , insol. cold.

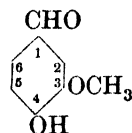
*Acetyl*: needles from EtOH. Aq. M.p. 145–6°.

*Benzoyl*: plates from EtOH. Aq. M.p. 178°.

Sabalitsehka, Tietz, *Chem. Zentr.*, 1932, I, 1110.

Zeltner, Landau, D.R.P., 258,887, (*Chem. Zentr.*, 1913, I, 1641).

**Vanillin** (*Protocatechuic aldehyde 3-methyl ether, 4-hydroxy-3-methoxybenzaldehyde*)



$\text{C}_8\text{H}_8\text{O}_3$

MW, 152

Widely occurring in plant world. Constituent of fruit of *Vanilla planifolia*, etc. Needles with characteristic odour from  $\text{H}_2\text{O}$  or ligroin. M.p. 80–1°. B.p. 170°/15 mm., 140–5°/6 mm. Very sol. EtOH,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CS}_2$ , AcOH, Py. Mod. sol. hot ligroin,  $\text{C}_6\text{H}_6$ . Insol. cold ligroin. Sol. 90 parts  $\text{H}_2\text{O}$  at 14°, 20 parts at 75°. Sublimes. Heat of comb.  $\text{C}_p$  9147 Cal.,  $\text{C}_v$  9144 Cal. Reacts in sol. as monobasic acid. Slowly oxidises in moist air  $\rightarrow$  vanillic acid. KOH fusion  $\rightarrow$  protocatechuic acid. NaHg or catalytic reduction  $\rightarrow$  vanillyl alcohol. Electrolytic reduction  $\rightarrow$  4-hydroxy-3-methoxytoluene.  $\text{FeCl}_3 \rightarrow$  bluish-violet col. Conc.  $\text{H}_2\text{SO}_4$  + 1-naphthol  $\rightarrow$  bluish-red col. Conc.  $\text{H}_2\text{SO}_4$  + 2-naphthol  $\rightarrow$  emerald-green col. Bromine  $\rightarrow$  red col. or red ppt. Zn and Mg salts spar. sol.  $\text{H}_2\text{O}$ . Used for flavouring and in perfumery.

*Me ether*: see Veratric Aldehyde.

*Et ether*:  $\text{C}_{10}\text{H}_{12}\text{O}_3$ . MW, 180. Prisms. M.p. 64–5°. Sublimes. Sol. EtOH,  $\text{Et}_2\text{O}$ . Spar. sol. hot  $\text{H}_2\text{O}$ .

*Propyl ether*:  $\text{C}_{11}\text{H}_{14}\text{O}_3$ . MW, 194. Cryst. from EtOH. Aq. M.p. 59–60°. *Semicarbazone*: needles from EtOH. M.p. 156°.

*Isopropyl ether*: pale yellow viscous liq. B.p. 150–2°/13 mm. *Semicarbazone*: needles from EtOH. M.p. 151–2°.

*2:4-Dinitrophenyl ether*:  $\text{C}_{14}\text{H}_{10}\text{O}_7\text{N}_2$ . MW, 318. Needles from AcOH. Aq. M.p. 131°.

*Picryl ether*:  $\text{C}_{14}\text{H}_9\text{O}_9\text{N}_3$ . MW, 363. Plates from  $\text{Et}_2\text{O}$ –ligroin. M.p. 114–16°.

*Benzyl ether*:  $\text{C}_{15}\text{H}_{14}\text{O}_3$ . MW, 242. Needles or plates from EtOH. M.p. 64–5°.

*p-Nitrobenzyl ether*:  $\text{C}_{15}\text{H}_{13}\text{O}_5\text{N}$ . MW, 287. Cryst. from EtOH. Aq. M.p. 124–5°.

*Acetyl*: needles from  $\text{Et}_2\text{O}$ . M.p. 102–3°.

*Benzoyl*: needles from EtOH. Aq. M.p. 78°.

*p-Toluenesulphonyl*: needles from EtOH. M.p. about 115°.

*2-Naphthalenesulphonyl*: m.p. 98°.

*Phenylurethane*: m.p. 116–17°.

*Oxime*: plates or needles from  $\text{H}_2\text{O}$ . M.p. 121–2°. Very sol. hot  $\text{H}_2\text{O}$ , EtOH,  $\text{Et}_2\text{O}$ . Insol.  $\text{C}_6\text{H}_6$ , ligroin. *BHCl*: m.p. 139°. *N-Acetyl*: needles from EtOH. Aq. M.p. 114°. *O:N-Diacetyl*: pale red needles. M.p. 95°.

**Azine**: yellow prisms from EtOH. M.p. 195–7°, solidifies and remelts at 230–5°.

**Anil**: slightly yellow needles from EtOH. M.p. 152–3°.

**Semicarbazone**: needles. M.p. 230°.

**Thiosemicarbazone**: needles. Sinters at 194°, m.p. 196–7°.

**Phenylhydrazone**: plates from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 105°.

**p-Bromophenylhydrazone**: yellow plates from EtOH. M.p. 146°.

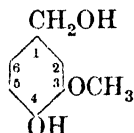
**p-Nitrophenylhydrazone**: red plates from AcOH. M.p. 227°.

**2:4-Dinitrophenylhydrazone**: orange-red micro-prisms from AcOH. M.p. 267–8°.

Mottern, *J. Am. Chem. Soc.*, 1934, **56**, 2107.

Hoffmann-La Roche, E.P., 399,723, (*Chem. Zentr.*, 1934, I, 127), D.R.P., 580,981, (*Chem. Zentr.*, 1933, II, 1763).

**Vanillyl Alcohol (4-Hydroxy-3-methoxybenzyl alcohol)**



C<sub>8</sub>H<sub>10</sub>O<sub>3</sub>

MW, 154

Prisms from H<sub>2</sub>O. M.p. 115°. Not distillable at ord. press. Sol. warm H<sub>2</sub>O, EtOH, Et<sub>2</sub>O.

**4-Me ether**: see Veratryl Alcohol.

**4-Et ether**: C<sub>10</sub>H<sub>14</sub>O<sub>3</sub>. MW, 182. Needles from EtOH. M.p. 56–7°. B.p. 185–7°/8 mm. Sol. Et<sub>2</sub>O, EtOH, AcOEt, C<sub>6</sub>H<sub>6</sub>. **Allophanate**: m.p. 173°. **Acetyl**: m.p. 22–3°. B.p. 170–3°/13 mm. **Benzoyl**: m.p. 49°.

**4-Acetyl**: m.p. 51°. B.p. 194–6°/13 mm.

**Diacetyl**: plates from C<sub>6</sub>H<sub>6</sub>-ligroin. M.p. 48°. B.p. 185°/12 mm.

**4-Benzoyl**: cryst. from AcOEt–EtOH. M.p. 90° (99°).

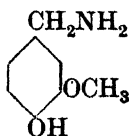
**Dibenzoyl**: m.p. 121°.

Goethals, *Chem. Zentr.*, 1937, I, 580.

Carothers, Adams, *J. Am. Chem. Soc.*, 1924, **46**, 1675.

Vavon, *Ann. chim.*, 1914, **1**, 159.

**Vanillylamine (4-Hydroxy-3-methoxybenzylamine)**



C<sub>8</sub>H<sub>11</sub>O<sub>2</sub>N

MW, 153

Dict. of Org. Comp.—III.

Needles + 2H<sub>2</sub>O. M.p. anhyd. 137° (145–6°). Becomes yellow in daylight. Easily decomp. with boiling H<sub>2</sub>O or alkalis.

**B,HCl**: m.p. 227°.

**N-Acetyl**: cryst. from AcOEt. M.p. 109–109.5°.

**N-Propionyl**: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 111.5°.

**N-Butyryl**: cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 75.5–76.5°.

**N-Valeryl**: cryst. from C<sub>6</sub>H<sub>6</sub>–pet. ether. M.p. 60–60.5°.

Ford-Moore, Phillips, *Rec. trav. chim.*, 1934, **53**, 854.

I.G., D.R.P., 442,774, F.P., 610,830, (*Chem. Zentr.*, 1927, II, 506); U.S.P., 1,873,402, (*Chem. Abstracts*, 1932, **26**, 5965).

Jones, Pyman, *J. Chem. Soc.*, 1925, 2592.

Nelson, *J. Am. Chem. Soc.*, 1919, **41**, 1118.

### Varianose

(C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>)<sub>n</sub>

MW, (162)<sub>n</sub>

White amorph. powder. Mod. sol. H<sub>2</sub>O. [α]<sub>D</sub><sup>20</sup> + 15° in H<sub>2</sub>O. Reduces hot Fehling's slightly. Gives no col. with I sol. Hyd. by min. acids → *d*-galactose (70%) + *d*-glucose (14%) + either *d*-idose or *l*-altrose (14%).

**Fully acetylated deriv.** ("triacetyl"): white powder. M.p. 148–55°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>, hot EtOH. Does not reduce Fehling's. [α]<sub>D</sub><sup>20</sup> + 30° in CHCl<sub>3</sub>, + 38.2° in Me<sub>2</sub>CO.

**Fully methylated deriv.** ("trimethyl"): white powder. M.p. 90–100°. Sol. cold, insol. hot H<sub>2</sub>O. Sol. CHCl<sub>3</sub>, Me<sub>2</sub>CO. [α]<sub>D</sub><sup>22</sup> + 15° in H<sub>2</sub>O, + 20° in CHCl<sub>3</sub>, + 23° in C<sub>6</sub>H<sub>6</sub>.

Haworth, Raistrick, Stacey, *Biochem. J.*, 1935, **29**, 2668.

### Vasicine.

See Poganine.

### Vellosine

C<sub>23</sub>H<sub>28</sub>O<sub>4</sub>N<sub>2</sub>

MW, 396

Constituent of bark of *Geissospermum Vellosii*. Prisms from EtOH. M.p. 189°. Sol. common org. solvents. Insol. H<sub>2</sub>O. [α]<sub>D</sub> + 22.8° in CHCl<sub>3</sub>.

**B,HCl**: cryst. + H<sub>2</sub>O. M.p. 180° decomp., anhyd. 245–8°.

**B,HBr**: needles + H<sub>2</sub>O. M.p. 194–5°.

**B,HI**: plates + H<sub>2</sub>O from EtOH.Aq. M.p. 217–18°.

**B,HNO<sub>3</sub>**: plates + H<sub>2</sub>O from EtOH.Aq. Decomp. at 225°.

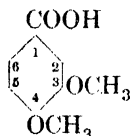
**B,H<sub>2</sub>SO<sub>4</sub>**: cryst. + H<sub>2</sub>O from EtOH.Aq. M.p. 210°.

$B_2H_2PtCl_6$ : cryst. M.p. about  $80^\circ$ .

Methiodide: cryst. from  $H_2O$ . M.p.  $264^\circ$ .

Freund, Fauvet, *Ann.*, 1894, **282**, 247.

**Veratric Acid** (Protocatechuic acid dimethyl ether, 3:4-dimethoxybenzoic acid)



$C_9H_{10}O_4$

MW, 182

Cryst. from  $H_2O$ . M.p. anhyd.  $181^\circ$ . Sublimes in rhombic cryst. Sol. 2,100 parts  $H_2O$  at  $14^\circ$ , 160 parts at  $100^\circ$ . Very sol. EtOH,  $Et_2O$ .  $k = 3.6 \times 10^{-5}$  at  $25^\circ$ . Ba salt spar. sol.  $H_2O$ .

Me ester:  $C_{10}H_{12}O_4$ . MW, 196. Needles from EtOH.Aq. M.p.  $59-60^\circ$ . B.p.  $300^\circ$  ( $283^\circ$ ). Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

Et ester:  $C_{11}H_{14}O_4$ . MW, 210. Needles from EtOH. M.p.  $43-4^\circ$ . B.p.  $295-6^\circ$ . Sol. EtOH,  $Et_2O$ . Insol.  $H_2O$ .

Chloride:  $C_9H_9O_3Cl$ . MW, 200.5. Cryst. M.p.  $70^\circ$ . B.p. about  $290^\circ$ .

Amide:  $C_9H_{11}O_3N$ . MW, 181. Cryst. M.p.  $164^\circ$ . Sol. hot  $H_2O$ . N-Di-Me:  $C_{11}H_{15}O_3N$ . MW, 209. M.p.  $103^\circ$ . B.p.  $203^{5/12}$  mm. N-Di-Et:  $C_{13}H_{19}O_3N$ . MW, 237. B.p.  $205^{10/12}$  mm.

Nitrile:  $C_9H_9O_2N$ . MW, 163. Needles from  $H_2O$ . M.p.  $67-8^\circ$ .

Anhydride:  $C_{18}H_{18}O_7$ . MW, 346. Prisms from AcOEt or  $C_6H_6$ . M.p.  $124-5^\circ$ .

Anilide: needles from EtOH. M.p.  $154^\circ$ .

Buck, Ide, *Organic Syntheses*, 1935, XV, 85.

Grignard, *Compt. rend.*, 1934, **198**, 625.

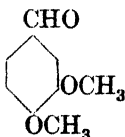
Rodionov, Fedorova, *Arch. Pharm.*, 1933, **271**, 287.

Tiemann, Matsumoto, *Ber.*, 1876, **9**, 937.

#### o-Veratric Acid.

See 2:3-Dimethoxybenzoic Acid.

**Veratric Aldehyde** (3:4-Dimethoxybenzaldehyde, protocatechuic aldehyde dimethyl ether, vanillin methyl ether, veratraldehyde)



$C_9H_{10}O_3$

MW, 166

Needles from  $Et_2O$ , ligroin,  $CCl_4$  or toluene. M.p.  $58^\circ$  ( $42^\circ$ ). B.p.  $285^\circ$ ,  $154-5^\circ/10$  mm. Sol. EtOH,  $Et_2O$ . Insol. cold  $H_2O$ . Readily oxidises in air.

Oxime: cryst. from ligroin. M.p.  $94-5^\circ$ . Sol. common org. solvents.  $BHCl$ : dark red solid. M.p.  $151^\circ$ .

Anil: cryst. from EtOH. M.p.  $81^\circ$ .

Azine: yellow plates from EtOH- $CHCl_3$ . M.p.  $169-70^\circ$ .

Semicarbazone: m.p.  $177^\circ$ .

Phenylhydrazone: yellow plates from EtOH. M.p.  $121^\circ$ .

2:4-Dinitrophenylhydrazone: orange prisms from AcOEt. M.p.  $264-5^\circ$ .

Johnson, Stevenson, *Organic Syntheses*, 1936, XVI, 91.

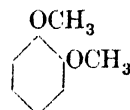
Briner, Tscherner, Paillard, *Helv. Chim. Acta*, 1925, **8**, 406.

Gatterman, *Ann.*, 1907, **357**, 367.

#### Veratrine.

See Cevadine.

**Veratrol** (Catechol dimethyl ether, 1:2-dimethoxybenzene)



$C_8H_{10}O_2$

MW, 138

Cryst. from pet. ether. M.p.  $22.5^\circ$ . B.p.  $206^{10}/759$  mm.  $D_4^{25}$  1.0842.  $n_D^{21.2}$  1.52870.

Picrate: red plates. M.p.  $56-7^\circ$ .

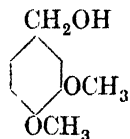
Voss, Blaneke, *Ann.*, 1931, **485**, 279.

Perkin, Weizmann, *J. Chem. Soc.*, 1906, **89**, 1649.

#### 2-Veratrolylcinchomeronic Acid.

See Papaveric Acid.

**Veratryl Alcohol** (3:4-Dimethoxybenzyl alcohol)



$C_9H_{12}O_3$

MW, 168

Viscous oil. B.p.  $296-7^\circ/732$  mm.,  $172^\circ/12$  mm.  $D_{17}^{25}$  1.179.  $n_D^{17}$  1.555. Sol.  $H_2O$ , EtOH.

Acetyl: viscous oil. B.p.  $170^\circ/12$  mm.  $D_4^{17}$  1.157.  $n_D^{17}$  1.5245.

Benzoyl: m.p.  $36-7^\circ$ . B.p.  $233-6^\circ/12$  mm.  $D_4^{19}$  1.193.  $n_D^{19}$  1.575.

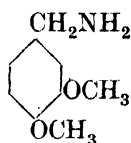
Phenylurethane: m.p.  $118^\circ$ .

Davidson, Bogert, *J. Am. Chem. Soc.*, 1935, **57**, 905.

Tiffeneau, Fuhrer, *Bull. soc. chim.*, 1914, **15**, 171.

Vavon, *Compt. rend.*, 1912, **154**, 360.

Tiffeneau, *Bull. soc. chim.*, 1911, **9**, 929.

**Veratrylamine** (3 : 4-Dimethoxybenzylamine) $C_9H_{13}O_2N$ 

MW, 167

Liq. B.p. 154–8°/12 mm.  $D_0^{20}$  1.143. Slowly volatile in steam.

*B,HCl*: cryst. from EtOH. M.p. 257–8°.

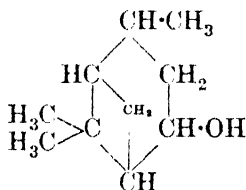
*Picrate*: m.p. 169°.

*Methiodide*: m.p. 228°.

Juliusberg, *Ber.*, 1907, **40**, 120.

Nelson, *J. Am. Chem. Soc.*, 1919, **41**, 1117.

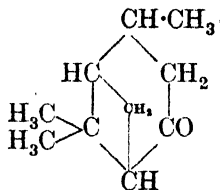
Douetteau, *Bull. soc. chim.*, 1911, **9**, 937.

**Verbanol** $C_{10}H_{18}O$ 

MW, 154

Needles from EtOH.Aq. or ligroin. M.p. 58°. B.p. 218°, 102°/20 mm.  $D_0^{20}$  0.940.  $n_D^{20}$  1.47018.  $[\alpha]_D^{20} + 24.4^\circ$ .

Blumann, Zeitschel, *Ber.*, 1913, **46**, 1191.

**Verbanone** $C_{10}H_{16}O$ 

MW, 152

B.p. 222°, 117°/32 mm.  $D_0^{20}$  0.961.  $n_D^{20}$  1.47518.  $[\alpha]_D^{20} + 52.5^\circ$ .

*Oxime*: plates from ligroin. M.p. 88° (77–8°). B.p. 148–60°/35 mm.  $[\alpha]_D^{20} - 30.5^\circ$ . *B,HCl*: prisms. M.p. 142° (rapid heat.).

*Semicarbazone*: cryst. from EtOH.Aq. De-comp. at 230°.

*Hydrazone*: m.p. 27°. B.p. 146°/22 mm.  $D_4^{27}$  0.986.  $n_D^{27}$  1.51734.  $[\alpha]_D^{27} - 29.2^\circ$ .

Wienhaus, Schumm, *Ann.*, 1924, **439**, 35.

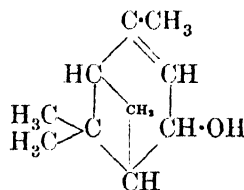
Blumann, Zeitschel, *Ber.*, 1913, **46**, 1192.

**Verbascose** $C_{36}H_{62}O_{31}$ 

MW, 990

Occurs in roots of mullein (*Verbascum thapsus*). Needles. M.p. 219–20°.  $[\alpha]_D^{20} + 169.9^\circ$  in  $H_2O$ . Does not reduce Fehling's. Hyd.  $\rightarrow$  glucose + fructose + galactose.

Bourquelot, Bridel, *Compt. rend.*, 1910, **151**, 760.

**Verbenol** $C_{10}H_{16}O$ 

MW, 152

*d.*

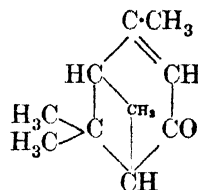
Liq. Cryst. in plates on cooling. M.p. 8°. B.p. 216–18° (with dehydration), 95°/9 mm.  $D_0^{20}$  0.9702.  $n_D^{20}$  1.4890.  $[\alpha]_D^{20} + 132.3^\circ$ .

*l.*

Liq. B.p. 100–4°/12 mm.

Blumann, Schmidt, *Ann.*, 1927, **453**, 48.

Blumann, Zeitschel, *Ber.*, 1913, **46**, 1195.

**Verbenone** $C_{10}H_{14}O$ 

MW, 150

*d.*

Constituent of Spanish verbena oil. M.p. 6.5°. B.p. 227–8°, 103–4°/16 mm. Sol.  $H_2O$ .  $D_0^{20}$  0.9976.  $n_D^{20}$  1.49928.  $[\alpha]_D^{20} + 249.6^\circ$ .

*Oxime*: cryst. from ligroin. M.p. 119–20°. B.p. 140–2°/17 mm.  $[\alpha]_D^{20} + 80^\circ$  in  $Et_2O$ .

*Semicarbazone*: plates from EtOH. M.p. 208–9°.  $[\alpha]_D^{20} + 77.6^\circ$ .

*l.*

Liq.  $D_0^{20}$  0.982.  $n_D^{20}$  1.4994.  $[\alpha]_D^{20} - 144^\circ$ .

*Semicarbazone*: m.p. 185–90°.

*dl.*

*Semicarbazone*: m.p. 180–1°.

Dupont, Zacharewicz, *Bull. soc. chim.*, 1935, **2**, 533.

Wienhaus, Schumm, *Ann.*, 1924, **439**, 20.

Blumann, Zeitschel, *Ber.*, 1913, **46**, 1188.

Kerschbaum, *Ber.*, 1900, **33**, 885.



*Et ester*:  $C_7H_{10}O_2$ . MW, 126. B.p.  $70-1^\circ/31$  mm.,  $57.2-57.5^\circ/13$  mm.  $D_4^{25.3}$  0.9348.  $n_D^{19.8}$  1.49306.

*Nitrile*:  $C_5H_5N$ . MW, 79. B.p.  $135-8^\circ$  (with polymerisation),  $48-50^\circ/28$  mm.  $D_4^{20}$  0.8644.  $n_D^{20}$  1.4880.

Coffmann, *J. Am. Chem. Soc.*, 1935, **57**, 1982.

Muskat, Becker, Lowenstein, *J. Am. Chem. Soc.*, 1930, **52**, 329.

Burton, Ingold, *J. Chem. Soc.*, 1929, 2028.  
Noffbohm, *Ann.*, 1917, **412**, 73.

### Vinyl Alcohol (*Ethenol*)



$C_2H_4O$  MW, 44

Does not exist in free state. Polymers of the alcohol and its esters used in synthetic resins.

*Acetyl*: vinyl acetate. Polymerises in air to transparent mass, polyvinyl acetate. Used in lacquers and resins.

*Benzoyl*: b.p.  $203^\circ$ .  $D^{20}$  1.065. Polymers used in lacquers.

*Et ether*: see Ethyl vinyl Ether.

*Vinyl ether*: see Divinyl Ether.

*Phenyl ether*:  $C_8H_8O$ . MW, 120. B.p.  $158-60^\circ$ .

I.G., D.R.P., 604,640, (*Chem. Zentr.*, 1935, II, 2125).

Evans, Looker, *J. Am. Chem. Soc.*, 1921, **43**, 1925.

Chem. Fabr. Griesheim-Elektron, D.R.P., 271,381, (*Chem. Zentr.*, 1914, I, 1316).

### Vinylallylcarbinol (1 : 5-Hexadienol-3)



$C_6H_{10}O$  MW, 98

Liq. B.p.  $133-4^\circ$ .  $D_4^{25}$  0.8596.  $n_D^{25}$  1.4464.

Levene, Haller, *J. Biol. Chem.*, 1929, **83**, 185.

### sym.-Vinylallylethylene.

See 1 : 3 : 6-Heptatriene.

### Vinylamine.

See Ethyleneimine.

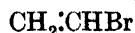
### Vinylanisole.

See under Hydroxystyrene.

### Vinylbenzene.

See Styrene.

### Vinyl bromide (*Bromoethylene*)



$C_2H_3Br$  MW, 107

F.p.  $-137.8^\circ$ . B.p.  $15.8^\circ$ .  $D_4^{11}$  1.5286.  $n_D$  1.4462.  $H_2O + PbO \longrightarrow$  acetylene.  $KMnO_4$

$\longrightarrow H \cdot COOH$ . Polymerises rapidly in sunlight  
 $\longrightarrow$  mixture of polymers.

Staudinger, Brunner, Feisst, *Helv. Chim. Acta*, 1930, **13**, 805.

Juvala, *Ber.*, 1930, **63**, 1991.

Bauer, U.S.P., 1,414,852, (*Chem. Abstracts*, 1922, **16**, 2150).

### 2-Vinylbutane.

See 3-Methyl-1-pentene.

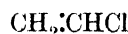
### Vinyl-n-butyl Alcohol.

See 1-Hexenol-6.

### Vinylbutylene.

See 1 : 3-Hexadiene and 3-Methyl-1 : 3-pentadiene.

### Vinyl chloride (*Chloroethylene*)



$C_2H_3Cl$  MW, 62.5

Gas. Liquefies on cooling. F.p.  $-159.7^\circ$ . B.p.  $-14^\circ$ . Heat of comb.  $C_p$  286.2 Cal. Polymerises in light or in absence of light in presence of catalysts.

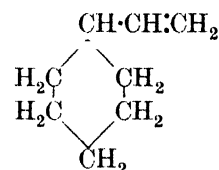
Carbide and Carbon Chem. Corp., U.S.P., 1,934,824, (*Chem. Abstracts*, 1934, **28**, 488); U.S.P., 1,926,638, (*Chem. Abstracts*, 1933, **27**, 5756).

Dana, Burdick, Jenkins, *J. Am. Chem. Soc.*, 1927, **49**, 2801.

### Vinyl cyanide.

See under Acrylic Acid.

### Vinylcyclohexane (*Cyclohexylethylene*)

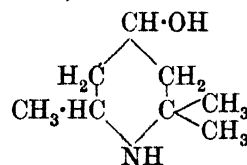


$C_8H_{14}$  MW, 110

Liq. B.p.  $130-1^\circ/749$  mm.  $D_4^{18}$  0.8166.  $n_D^{19}$  1.455.

Lewina, Zurikow, *Chem. Zentr.*, 1936, I, 4289.

### Vinyl diacetonealkamine (4-Hydroxy-2 : 2 : 6-trimethylpiperidine)



$C_8H_{17}ON$  MW, 143

$\alpha$ -Form :

*d*-.  
*B, HCl*: does not melt below 300°.  $[\alpha]_D$   
 + 13.3° in H<sub>2</sub>O.

*l*-.  
 Prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 79–81°.  $[\alpha]_{5461}$   
 – 18.5° in H<sub>2</sub>O.

*Picrate*: needles. M.p. 242–4°.

*dl*-.  
 Cryst. from C<sub>6</sub>H<sub>6</sub>. M.p. 137–8°. B.p. 209–  
 11°. Sol. 9 parts boiling H<sub>2</sub>O. Spar. sol. pet.  
 ether.

*B, HCl*: prisms from EtOH.Aq.  
*N-Me*: C<sub>9</sub>H<sub>19</sub>ON. MW, 157. Pale yellow  
 oil. B.p. 218–20°. Very sol. H<sub>2</sub>O. *Dihydrate*:  
 plates from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 39–40°.

*B, HCl*: m.p. 192°. *B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: cryst. M.p.  
 208° decomp. *O-Benzoyl*: pale yellow oil.  
 B.p. 194–5°/16 mm. *Picrate*: cryst. from  
 EtOH.Aq. M.p. 180–1°.

*N-Nitroso*: C<sub>8</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>. MW, 172. Cryst.  
 from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 92°.

*O-Benzoyl*: see  $\beta$ -Eucaine.

*Picrate*: plates from H<sub>2</sub>O. M.p. 171–2°.

$\beta$ -Form :

*d*-.  
 Plates from C<sub>6</sub>H<sub>6</sub>. M.p. 121–3°.

*B, HCl*: plates. M.p. 217–19°.  $[\alpha]_{5461}$  + 22.85°.  
*Picrate*: needles from H<sub>2</sub>O. M.p. 181–2°.

*dl*-.  
 Prisms from C<sub>6</sub>H<sub>6</sub>. M.p. 160–1°. B.p. 204–5°.

Sol. 30 parts boiling C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O,  
 Et<sub>2</sub>O. Na amylate in amyl alcohol  $\rightarrow$  *dl*- $\alpha$ -  
 form.

*N-Me*: prisms from pet. ether. M.p. 70–2°.  
 B.p. 220°/744 mm. Very sol. H<sub>2</sub>O. Na amyl-  
 ate in amyl alcohol  $\rightarrow$  *dl*- $\alpha$ -*N-Me*. *B, HCl*:  
 m.p. 58°. *Nitrate*: prisms. M.p. 163° decomp.  
*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: cryst. M.p. 218° decomp. *Meth-*  
*iodide*: prisms from EtOH. Decomp. at 270°.  
*O-Benzoyl*: pale yellow oil. B.p. 195°/15 mm.  
*Picrate*: yellow cryst. from EtOH.Aq. M.p.  
 213°.

*N-Nitroso*: yellow needles from C<sub>6</sub>H<sub>6</sub>-pet.  
 ether. M.p. 60°.

*O-Benzoyl*: see Iso- $\beta$ -eucaine.

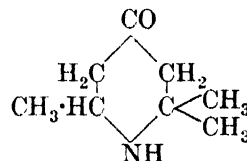
*Picrate*: plates from H<sub>2</sub>O. M.p. 171–2°.

King, *J. Chem. Soc.*, 1924, 125, 41.

Harries, Zart, *Ann.*, 1918, 417, 176.

Harries, *Ann.*, 1897, 294, 372; 1897, 296,  
 334.

**Vinyl diacetoneamine** (4-Keto-2 : 2 : 6-tri-  
 methylpiperidine, 2 : 2 : 6-trimethyl- $\gamma$ -piperidone)



C<sub>8</sub>H<sub>15</sub>ON

MW, 141

Hygroscopic plates and prisms. M.p. 27°.  
 B.p. 199–200°. NaHg  $\rightarrow$  vinyl diacetonealk-  
 amine.

*B<sub>2</sub>H<sub>2</sub>SO<sub>4</sub>*: needles from EtOH.Aq. M.p.  
 above 105° decomp.

*Nitrate*: needles from Me<sub>2</sub>CO-pet. ether.  
 M.p. 160–1°.

*B<sub>2</sub>(COOH)<sub>2</sub>*: m.p. 184–5° decomp.

*N-Me*: C<sub>9</sub>H<sub>17</sub>ON. MW, 155. Oil. B.p.  
 96–7°/14 mm. *Oxime*: prisms from pet. ether.  
 M.p. 93°.

*N-Nitroso*: C<sub>8</sub>H<sub>14</sub>O<sub>2</sub>N<sub>2</sub>. MW, 170. Pale yellow  
 plates from MeOH.Aq. M.p. 61°. *Oxime*: cryst.  
 from H<sub>2</sub>O. M.p. 195°.

*N-Acetyl*: prisms from Et<sub>2</sub>O. M.p. 92°.

*N-m-Nitrobenzoyl*: needles from Me<sub>2</sub>CO. M.p.  
 159–60°.

*N-p-Nitrobenzoyl*: needles from Me<sub>2</sub>CO. M.p.  
 170°.

*Oxime*: plates from EtOH. M.p. 151°. Sol.  
 5 parts boiling H<sub>2</sub>O. *N-Acetyl*: cryst. from  
 EtOH. M.p. 130°.

*Semicarbazone*: cryst. from EtOH-C<sub>6</sub>H<sub>6</sub>. M.p.  
 196–7°.

*Picrate*: prisms from H<sub>2</sub>O. M.p. 198–9°.

Harries, Schellhorn, *Ann.*, 1918, 417, 131.

Harries, *Ber.*, 1896, 29, 522.

$\alpha$ -Vinyl diphenylmethane.

See 3 : 3-Diphenylpropylene.

**Vinyl fluoride** (Fluoroethylene)



C<sub>2</sub>H<sub>3</sub>F

MW, 46

Gas at ord. temps. B.p. –51°. Burns with  
 green flame. Sol. EtOH, Me<sub>2</sub>CO. Insol. H<sub>2</sub>O.

Swarts, *Bull. soc. chim.*, 1919, 25, 163;

*Chem. Zentr.*, 1909, II, 1414; 1901, II,  
 804.

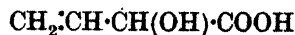
**Vinylformic Acid.**

See Acrylic Acid.

**Vinylfuran.**

See  $\alpha$ -Furylethylene.

**Vinylglycollic Acid** (1-Hydroxyvinylacetic  
 acid)



C<sub>4</sub>H<sub>6</sub>O<sub>3</sub>

MW, 102

Hygroscopic needles. M.p. 42–4°. B.p. 128.6–130.2°/12–13 mm. Very sol. H<sub>2</sub>O. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Insol. CS<sub>2</sub>. Volatile in steam.  $k = 4.6 \times 10^{-4}$ . Min. acids  $\rightarrow$  1-ketobutyric acid.

*Et ester*: C<sub>6</sub>H<sub>10</sub>O<sub>3</sub>. MW, 130. Liq. with pleasant odour. B.p. 173° slight decomp., 68°/15 mm. D<sub>4</sub><sup>16</sup> 1.0470. n<sub>D</sub><sup>13</sup> 1.436. *Acetyl*: b.p. 89°/15 mm. D<sub>4</sub><sup>16</sup> 1.055. n<sub>D</sub><sup>16</sup> 1.429.

*Amide*: C<sub>4</sub>H<sub>7</sub>O<sub>2</sub>N. MW, 101. Plates from Et<sub>2</sub>O. M.p. 80–8°. B.p. 155–8°/20–1 mm. slight decomp. Sol. H<sub>2</sub>O, EtOH. Spar. sol. Et<sub>2</sub>O.

*Nitrile*: see under Acrolein.

Glattfield, Hoen, *J. Am. Chem. Soc.*, 1935, 57, 1406.

Kirmann, Rambaud, *Compt. rend.*, 1932, 194, 1169.

Sleen, *Rec. trav. chim.*, 1902, 21, 222.

### Vinylidene bromide.

See *unsym.*-Dibromoethylene.

### Vinylidene chloride.

See *unsym.*-Dichloroethylene.

### Vinylidene-ethyl Alcohol.

See 4-Hydroxy-1 : 2-butadiene.

### Vinyl iodide (Iodoethylene)

C<sub>2</sub>H<sub>3</sub>I      CH<sub>2</sub>:CHI      MW, 154

B.p. 56–56.5°. D<sub>20</sub> 2.037. n<sub>D</sub> 1.53845. De-comp. on exposure to ultra-violet light  $\rightarrow$  C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub> and I.

Spence, *J. Am. Chem. Soc.*, 1933, 55, 1290.

### Vinylisoamylcarbinol.

See 6-Methyl-1-heptenol-3.

### 1-Vinylisobutylene.

See 4-Methyl-1 : 3-pentadiene.

### Vinylisobutyric Acid.

See Dimethylvinylacetic Acid.

### 1-Vinylisopentane.

See 4-Methyl-1-hexene.

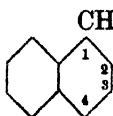
### sym.-Vinylisopropenylethane.

See 2-Methyl-1 : 5-hexadiene.

### Vinyl-naphthalene.

See Naphthylethylene.

**Vinyl-1-naphthylcarbinol** (1- $\alpha$ -Naphthyl-allyl alcohol, 1- $\alpha$ -hydroxyallylnaphthalene)



C<sub>13</sub>H<sub>12</sub>O

MW, 184

Viscous oil. B.p. 186–7°/19 mm.

*Phenylurethane*: needles from pet. ether. M.p. 108–9°.

*p-Nitrobenzoyl*: prisms from EtOH. M.p. 79–80°.

Burton, *J. Chem. Soc.*, 1931, 761.

**Vinyl-2-naphthylcarbinol** (1- $\beta$ -Naphthyl-allyl alcohol, 2- $\alpha$ -hydroxyallylnaphthalene).

Viscous oil. B.p. 195–8°/21 mm.

*Phenylurethane*: prisms from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 134–5°.

See previous reference.

### 2-Vinylpentane.

See 3-Methyl-1-hexene.

### 1-Vinyl-2-pentene.

See 1 : 4-Heptadiene.

### Vinylphenetole.

See under *p*-Hydroxystyrene.

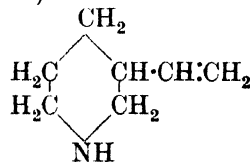
### Vinylphenol.

See Hydroxystyrene.

### Vinylphenylcarbinol.

See 1-Phenylallyl Alcohol.

**3-Vinylpiperidine** ( $\beta$ -Vinylpiperidine, 3-piperidylethylene)



C<sub>7</sub>H<sub>13</sub>N      MW, 111

Liq. B.p. 152–5°. D<sub>4</sub><sup>25</sup> 0.9274. n<sub>D</sub><sup>25</sup> 1.4731.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: cryst. + 2H<sub>2</sub>O from EtOH.Aq. M.p. 223–4° decomp.

N-*Me*: C<sub>8</sub>H<sub>15</sub>N. MW, 125. Liq. B.p. 161–2°/724 mm. Sol. 30 parts cold H<sub>2</sub>O. Spar. sol. hot H<sub>2</sub>O. Decolorises acid KMnO<sub>4</sub>. Zn + HCl  $\rightarrow$  1-methyl-3-ethylpiperidine. B<sub>2</sub>HAuCl<sub>4</sub>: prisms from H<sub>2</sub>O. M.p. 58–9°. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: orange-red plates or prisms. M.p. 185–6° decomp. *Picrate*: yellow prisms or needles. M.p. 193–4°.

N-*Et*: C<sub>9</sub>H<sub>17</sub>N. MW, 139. Liq. B.p. 185–90°/721 mm. Spar. sol. H<sub>2</sub>O. B<sub>2</sub>HAuCl<sub>4</sub>: cryst. M.p. 44–5°. B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: cryst. from H<sub>2</sub>O. M.p. 175–6° decomp.

Merchant, Marvel, *J. Am. Chem. Soc.*, 1928, 50, 1201.

Lipp, Widmann, *Ann.*, 1915, 409, 92; *Ber.*, 1905, 38, 2481.

### 4-[3-Vinylpiperidyl]-acetic Acid.

See Meroquinene.

### 2-Vinylpropane.

See 3-Methylbutylene-1.



**symt.-Vinylpropenylethylene.**

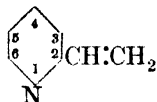
See 1 : 3 : 5-Heptatriene.

**2-Vinylpropionic Acid.**

See Allylacetic Acid.

**1-Vinyl-3-propylidene-propylene.**

See 1 : 3 : 5-Heptatriene.

**2-Vinylpyridine** ( $\alpha$ -Vinylpyridine, 2-pyridylethylene) $C_7H_7N$ 

MW, 105

Liq. B.p. 79–82°/29 mm.  $D_4^{20}$  0.9985. Very sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Spar. sol. H<sub>2</sub>O. Volatile in steam. KMnO<sub>4</sub> → picolinic acid. Na + EtOH → 2-ethylpiperidine.

$B_2HAuCl_4$ : yellow needles from H<sub>2</sub>O. M.p. 144°.

$B_2H_2PtCl_6$ : needles or plates. M.p. 174° decomp.

Löffler, Grosse, *Ber.*, 1907, **40**, 1326.

**4-Vinylpyridine** ( $\gamma$ -Vinylpyridine, 4-pyridylethylene).

Liq. B.p. 59°/12 mm.

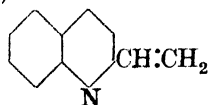
$B_2H_2PtCl_6$ : sinters at 200°, m.p. above 350°.

Picrate: yellow leaflets from C<sub>6</sub>H<sub>6</sub>. M.p. 197–8°.

Meisenheimer, *Ann.*, 1920, **420**, 208.

**Vinylpyridinium hydroxide.**

See Pyridine-neurine.

**2-Vinylquinoline** ( $\alpha$ -Vinylquinoline, 2-quinolyethylene) $C_{11}H_9N$ 

MW, 155

Pale yellow oil. Decomp. on dist. at 10 mm. Volatile in steam.

$B_2HgCl_2.HCl$ : needles from H<sub>2</sub>O. M.p. 151–2° decomp.

$B_2HAuCl_4$ : yellowish-red needles from dil. min. acids. M.p. 158–9°.

$B_2H_2PtCl_6$ : orange-red needles. Decomp. at 182°.

$B_2H_2PtCl_6.4H_2O$ : orange needles from dil. min. acids. M.p. 186–7°.

Methner, *Ber.*, 1894, **27**, 2691.

**Vinytoluene.**

See Methylstyrene.

**4-Vinylvaleric Acid.**

See 5-Heptenic Acid.

**Violanin** (*Rhamnoglucoside of delphinidin*) $C_{27}H_{28}O_{15}$ 

MW, 592

Constituent of *Viola tricolor*, Linn.

*Chloride*: bluish-violet plates with green metallic lustre. Sol. alkalis and alk. carbonates → blue sols. Alc. FeCl<sub>3</sub> → blue col. Boiling dil. HCl → delphinidin + rhamnose + glucose.

Willstätter, Weil, *Ann.*, 1916, **412**, 178.

**Violanthrone.**

See Dibenzanthrone.

**Violaquercitrin.**

See Rutin.

**Violaxanthin** $C_{40}H_{56}O_4$ 

MW, 600

Constituent of *Viola tricoloris*, etc. Red prisms from MeOH, EtOH–Et<sub>2</sub>O, or CS<sub>2</sub>. M.p. 208° (198–9°).  $[\alpha]_{D}^{20} + 35^\circ$  (+ 38°) in CHCl<sub>3</sub>. Optical maxima in CS<sub>2</sub> at 500.5, 469 and 440 m $\mu$ , in CHCl<sub>3</sub> at 482, 451.5 and 424 m $\mu$ . Sol. conc. H<sub>2</sub>SO<sub>4</sub> → indigo-blue col. Sol. H·COOH → blue col. Sol. cold AcOH → green col. SbCl<sub>3</sub> + CHCl<sub>3</sub> → deep blue col. Conc. HCl in AcOH → greenish-blue col.

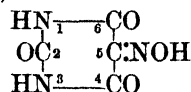
Zechmeister, Tuzson, *Ber.*, 1934, **67**, 824.

Kuhn, Grundmann, *ibid.*, 596.

Zechmeister, Cholnoky, *Z. physiol. Chem.*, 1932, **208**, 26.

Kuhn, Winterstein, *Ber.*, 1931, **64**, 326.

Karrer, Morf, *Helv. Chim. Acta*, 1931, **14**, 1044.

**Violuric Acid** (5-Isonitrosobarbituric acid, alloxan-5-oxime) $C_4H_3O_4N_3$ 

MW, 157

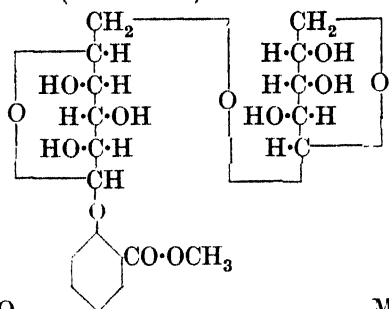
Rhombic cryst. Mod. sol. H<sub>2</sub>O → violet sol. Sol. EtOH.  $k = 2.7 \times 10^{-5}$  at 25°. FeCl<sub>3</sub> → blue col. Warm with HCl → NH<sub>2</sub>OH. Red. → uramil. Forms series of variously coloured metallic salts.

5-O-*Me ether*: C<sub>5</sub>H<sub>5</sub>O<sub>4</sub>N<sub>3</sub>. MW, 171. Plates from ligroin. M.p. 268°. Sol. H<sub>2</sub>O, alkalis and org. solvents → yellow sols.  $k = 1.8 \times 10^{-7}$  at 25°.

5-O-*Benzyl ether*: C<sub>11</sub>H<sub>9</sub>O<sub>4</sub>N<sub>3</sub>. MW, 247. Plates from C<sub>6</sub>H<sub>6</sub>. M.p. 226° decomp. Sol. H<sub>2</sub>O, EtOH.

Andreasch, *Monatsh.*, 1900, **21**, 286.

Hantzsch, Isherwood, *Ber.*, 1909, **42**, 986.

Violutin (*Violutoside*)C<sub>19</sub>H<sub>26</sub>O<sub>12</sub>

MW, 446

Occurs in *Viola cornuta*. M.p. 168.5° (173°).  $[\alpha]_D^{20} - 36.20^\circ$  in H<sub>2</sub>O,  $[\alpha]_{5461}^{21} - 39.72^\circ$  in H<sub>2</sub>O,  $[\alpha]_{5790}^{21} - 36.21^\circ$  in H<sub>2</sub>O. Sol. H<sub>2</sub>O. Prac. insol. dry Me<sub>2</sub>CO or AcOEt. Hyd. → methyl salicylate + *d*-glucose + *l*-arabinose.

Hexa-acetyl: m.p. 158°.  $[\alpha]_{5461}^{21} - 42.94^\circ$  in Me<sub>2</sub>CO.

Picard, *Compt. rend.*, 1926, **182**, 1167.

Robertson, Waters, *J. Chem. Soc.*, 1932, 2770.

## Violutoside.

See Violutin.

## Viscol

C<sub>30</sub>H<sub>50</sub>O

MW, 426

Isolated in two forms from mistletoe or cloves.

α-

Needles from MeOH or Me<sub>2</sub>CO.Aq. M.p. 200°.  $[\alpha]_D^{20} + 85.3^\circ$  in CHCl<sub>3</sub>.

Acetyl deriv.: needles from EtOH. M.p. 241°.  $[\alpha]_D^{20} + 80.2^\circ$  in CHCl<sub>3</sub>.

Benzoyl deriv.: plates from EtOH or Me<sub>2</sub>CO. M.p. 240°.

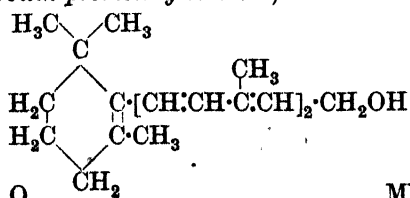
β-

Needles from Me<sub>2</sub>CO.Aq. or EtOH.Aq. M.p. 217°.  $[\alpha]_D^{20} + 55.7^\circ$  in CHCl<sub>3</sub>.

Acetyl deriv.: needles from EtOH or AcOEt-MeOH. M.p. 213°.  $[\alpha]_D^{20} + 42.8^\circ$  in CHCl<sub>3</sub>.

Benzoyl deriv.: plates from EtOH or Me<sub>2</sub>CO. M.p. 257°.

Bauer, Gerloff, *Arch. Pharm.*, 1936, **274**, 473.

Vitamin A (*Antixerophthalmic, anti-infective and growth-promoting vitamin*)C<sub>20</sub>H<sub>30</sub>O

MW, 286

Constituent of many fish-liver oils, milk, egg-yolk, seed embryos, etc. Pale yellow viscous liq. B.p. 137-8°/10<sup>-6</sup> mm. Sol. all org. solvents. Insol. H<sub>2</sub>O. Unstable in air. Characteristic absorption band at 328 mμ. SbCl<sub>3</sub> in CHCl<sub>3</sub> → blue sol. with absorption bands at 583 and 617 mμ. Conc. H<sub>2</sub>SO<sub>4</sub> → violet col. Physiological activity destroyed on reduction.

Karrer, Morf, Schöpp, *Helv. Chim. Acta*, 1931, **14**, 1431.

Heilbron *et al.*, *Biochem. J.*, 1932, **26**, 1178.

Hamano, *Sci. Papers Inst. Phys. Chem. Research, Tokyo*, 1937, **32**, 44.

Terada, *Chem. Abstracts*, 1934, **28**, 5182.

## Vitamin B.

Numerous factors, in addition to B<sub>1</sub> and B<sub>2</sub>, are described in the literature, but no chemically pure substances, other than those described as B<sub>1</sub> and B<sub>2</sub>, have been isolated. The following are representative references:

O'Brien, *Biochem. J.*, 1934, **28**, 926.

Kinnersley *et al.*, *Biochem. J.*, 1933, **27**, 225.

Keenan *et al.*, *J. Biol. Chem.*, 1933, **103**, 671.

Eddy, Gurin, Keresztesy, *J. Biol. Chem.*, 1930, **87**, 729.

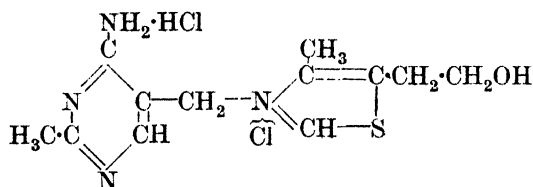
Carter, Kinnersley, Peters, *Biochem. J.*, 1930, **24**, 1832.

Reader, *Biochem. J.*, 1930, **24**, 77; 1929, **23**, 689.

Peters, *Nature*, 1929, **124**, 411.

Smith, *Chem. Abstracts*, 1928, **22**, 2399.

Vitamin B<sub>1</sub> (*Anti-beri-beri or antineuritic vitamin, aneurin, torulin, oryzanin, vitamin F, vitamin P*)

C<sub>12</sub>H<sub>18</sub>ON<sub>4</sub>Cl<sub>2</sub>S

MW, 337

Rice husks are chief source. Variable constituent of yeast, milk, green leaves, roots and tubers. Cryst. from MeOH-Et<sub>2</sub>O. M.p. 233-4°. Plates from MeOH-EtOH or H<sub>2</sub>O-EtOH. M.p. 250°. Very sol. H<sub>2</sub>O. Spar. sol. EtOH, Me<sub>2</sub>CO. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Generally more sol. than B<sub>2</sub>. Thermolabile and more readily decomp. by alkalis than B<sub>2</sub>. Sol. in H<sub>2</sub>O or

EtOH shows absorption bands at 235 mμ and 267 mμ.

*Nitrate*: m.p. 164–5°.

*Sulphate*: two forms. (i) M.p. 203°. (ii) M.p. 276–8°.

Todd, Bergel, *J. Chem. Soc.*, 1937, 364.

Williams *et al.*, *J. Am. Chem. Soc.*, 1937, 59, 216; 1936, 58, 1063; 1935, 57, 517, 536, 1093, 1751, 1849, 1856.

Itter, Orent, McCollum, *J. Biol. Chem.*, 1935, 108, 571.

Kinnersley, O'Brien, Peters, *Biochem. J.*, 1935, 29, 701, 716.

Kakefuda, *Chem. Abstracts*, 1935, 29, 6278.

Ohdake, *Chem. Zentr.*, 1935, I, 3677.

Windaus *et al.*, *Z. physiol. Chem.*, 1932, 204, 123.

Veen, *Z. physiol. Chem.*, 1932, 208, 125.

### Vitamin B<sub>2</sub>.

See Lactoflavine.

### Vitamin C,

See Ascorbic Acid.

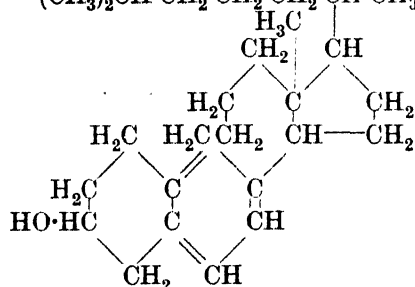
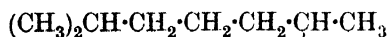
### Vitamin D<sub>1</sub>.

Has been shown to be a mixture of Vitamin D<sub>2</sub> and lumisterol.

### Vitamin D<sub>2</sub>.

See Calciferol.

### Vitamin D<sub>3</sub> (Anti-rachitic vitamin)



C<sub>27</sub>H<sub>44</sub>O

MW, 384

The vitamin D of fish-liver oils. Cryst. M.p. 82–4°.  $[\alpha]_D^{20} + 83.3^\circ$  in Me<sub>2</sub>CO. Shows absorption maximum at 265 mμ. As physiologically active as calciferol on rat test.

3:5-Dinitrobenzoyl: yellow needles. M.p. 129°.

Allophanate: cryst. from Me<sub>2</sub>CO. M.p. 173–4°.

Schenck, *Naturwissenschaften*, 1937, 10, 159.

Windaus, Schenck, Werder, *Z. physiol. Chem.*, 1936, 241, 100.

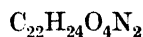
### Vitamin E.

See α-Tocopherol.

### Volemitol.

See Sedoheptitol.

### Vomicine



MW, 380

Strychnine alkaloid. Needles from EtOH.Aq. or EtOH-CHCl<sub>3</sub>, prisms from Me<sub>2</sub>CO. M.p. 278–80°.  $[\alpha]_D^{20} + 80.4^\circ$  in EtOH. Acid FeCl<sub>3</sub> → reddish-violet col. CrO<sub>3</sub> + H<sub>2</sub>SO<sub>4</sub> → deep red sol. HNO<sub>3</sub> → brown col. Hot MeOH-KOH → green sol.

B,HCl: prisms from H<sub>2</sub>O. M.p. 245° decomp.

Methosulphate: cryst. from MeOH.Aq. M.p. 264° decomp.

Me ether: C<sub>23</sub>H<sub>26</sub>O<sub>4</sub>N<sub>2</sub>. MW, 394. Needles from EtOH. M.p. 286–90° decomp.  $[\alpha]_D^{20} + 16.4^\circ$  in EtOH.

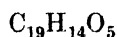
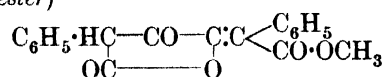
Benzylidene deriv.: yellow plates from EtOH-CHCl<sub>3</sub>. M.p. 280° decomp.

Wieland, Horner, *Ann.*, 1937, 528, 73.

Wieland, Calvet, *Ann.*, 1931, 491, 124.

Wieland, Oertel, *Ann.*, 1929, 469, 193.

### Vulpinic Acid (Vulpic acid, pulvinic acid methyl ester)



MW, 322

Occurs in many lichens. Exists in keto-form but can react as enol. Yellow plates from EtOH, needles or prisms from Et<sub>2</sub>O. M.p. 148–9°. Very sol. CHCl<sub>3</sub>. Mod. sol. Et<sub>2</sub>O. Spar. sol. boiling EtOH. Insol. boiling H<sub>2</sub>O. Hyd. → pulvinic acid. Reacts strongly acid and forms water stable salts. Poisonous.

Me ether: C<sub>20</sub>H<sub>16</sub>O<sub>5</sub>. MW, 336. Needles from EtOH. M.p. 143–4°.

Et ether: C<sub>21</sub>H<sub>18</sub>O<sub>5</sub>. MW, 350. Plates from EtOH. M.p. 138–9°.

Acetate: needles from EtOH or AcOH. M.p. 148° (156°).

Phenylacetate: needles from AcOH. M.p. 172°.

Benzoate: cryst. from AcOH. M.p. 176°.

Phenylurethane: yellow needles from EtOH. M.p. 237°.

Semicarbazone: yellow microcryst. M.p. 175°.

Asano, Kameda, *Ber.*, 1935, 68, 1569.

Koller, Pfeiffer, *Monatsh.*, 1933, 62, 164.

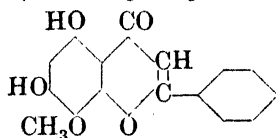
Karrer, Gehreckens, Heuss, *Helv. Chim. Acta*, 1926, 9, 456.

Mazza, *Chem. Zentr.*, 1926, II, 1037.

Volhard, *Ann.*, 1894, 282, 14.

Schenck, *ibid.*, 39.

## W

**Wogonin** (5 : 7-Dihydroxy-8-methoxyflavone) $C_{16}H_{12}O_5$ 

MW, 284

Present in small amount in roots of *Scutellaria baicalensis*, Georgi. Yellow needles from EtOH.Aq. M.p. 203°. Very sol. MeOH, EtOH. Sol. Et<sub>2</sub>O, AcOH, CHCl<sub>3</sub>, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O. Insol. CS<sub>2</sub>, ligroin.

*Mono-Me ether*: C<sub>17</sub>H<sub>14</sub>O<sub>5</sub>. MW, 298. M.p. 183°.

*Di-Me ether*: C<sub>18</sub>H<sub>16</sub>O<sub>5</sub>. MW, 312. Needles from H<sub>2</sub>O. M.p. 167-8°.

*Acetyl*: m.p. 152-3°.

*Benzoyl*: m.p. 170°.

Hattori, Hayashi, *J. Chem. Soc. Japan*, 1933, 54, 919.

Hattori, *Acta Phytochimica*, 1930, 5, 99.

**Wood sugar.**

See Xylose.

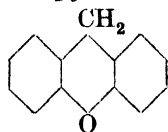
## X

**Xanthaline.**

See Papaveraldine.

**Xanthanoic Acid.**

See Xanthene-5-carboxylic Acid.

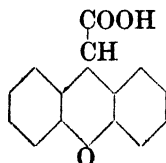
**Xanthene** (*Dibenzpyran*, *xanthane*) $C_{13}H_{10}O$ 

MW, 182

Leaflets from EtOH. M.p. 100.5° (98.5°). B.p. 310-12°. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, ligroin. Spar. sol. EtOH, AcOH, pet. ether. Insol. H<sub>2</sub>O. Sublimes slowly below m.p. Volatile in steam. Ox. → xanthone.

Ipatiev, Orlov, Petrov, *Ber.*, 1927, 60, 130.

Heller, v. Kostanecki, *Ber.*, 1908, 41, 1325.

**Xanthene-5-carboxylic Acid** (*Xanthanoic acid*) $C_{14}H_{10}O_3$ 

MW, 226

Needles from 50% EtOH. M.p. 223-4°.

*Me ester*: C<sub>15</sub>H<sub>12</sub>O<sub>3</sub>. MW, 240. Cryst. from MeOH. M.p. 85-6°.

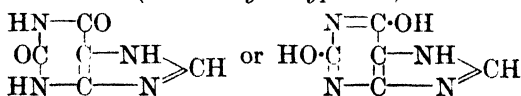
Conant, Garvey, *J. Am. Chem. Soc.*, 1927, 49, 2085.

**Xanthanol-5.**

See Xanthidrol.

**Xanthic Acid.**

See Xanthogenic Acid.

**Xanthine** (2 : 6-Dihydroxypurine) $C_5H_4O_2N_4$ 

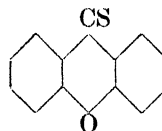
MW, 152

Found in potatoes, coffee beans, etc. Cryst. powder. Sol. 1400 parts H<sub>2</sub>O at 100°, 14,400 parts at 16°. Sol. 3000 parts EtOH at 17°. Very sol. KOH. Decomp. on heating. Very weak base. Conc. HCl at 220° → CO<sub>2</sub> + NH<sub>3</sub> + formic acid + glycine.

*Perchlorate*: cryst. + H<sub>2</sub>O. Sinters at 255°, m.p. 262-4° decomp.

Frèrejacque, *Compt. rend.*, 1930, 191, 950.

Biltz, Beck, *J. prakt. Chem.*, 1928, 118, 166.

**Xanthione** $C_{13}H_8OS$ 

MW, 212

Red needles from C<sub>6</sub>H<sub>6</sub>. M.p. 156°. Sol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. EtOH. Insol. H<sub>2</sub>O. Conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. with green fluor.

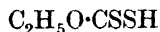
Schönberg, Schütz, Nickel, *Ber.*, 1928, 61, 1382.

Meyer, Szanecki, *Ber.*, 1900, 33, 2580.

**Xanthochelidonic Acid.**

See 1 : 3 : 5-Triketopimelic Acid.

**Xanthogenic Acid** (Dithiocarbonic O-ethyl ester, ethoxydithioformic acid, ethylxanthogenic acid, xanthic acid)



MW, 122

Unstable oily liq. F.p. about  $-53^\circ$ . Decomp. at  $25^\circ \rightarrow \text{CS}_2 + \text{C}_2\text{H}_5\text{OH}$ .

$\text{NH}_4$  salt: unstable powder.

$\text{K}$  salt: cryst. Sol.  $\text{H}_2\text{O}$ ,  $\text{EtOH}$ . Prac. insol.  $\text{Et}_2\text{O}$ .  $D^{21}_D$  1.558. Decomp. by boiling  $\text{H}_2\text{O}$  or by heat. Used as soil fumigant.

$\text{Me}$  ester:  $\text{C}_4\text{H}_8\text{OS}_2$ . MW, 136. B.p.  $182-3^\circ$ .  $D^{26}_D$  1.1189. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

$\text{Et}$  ester:  $\text{C}_5\text{H}_{10}\text{OS}_2$ . MW, 150. B.p.  $199-200^\circ$ ,  $76^\circ/10$  mm. Sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .  $D^{19}_D$  1.085.  $n^{18}_D$  1.5370.

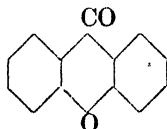
Anhydride: golden-yellow needles from  $\text{EtOH}$ . M.p.  $55^\circ$ . Very sol.  $\text{EtOH}$ . Sol.  $\text{Et}_2\text{O}$ . Insol.  $\text{H}_2\text{O}$ .

Willcox, *J. Am. Chem. Soc.*, 1906, **28**, 1032.

Raag, *Chem.-Ztg.*, 1910, **34**, 83.

Reillen, Elben, Everet, *Ann.*, 1931, **485**, 50.

Great Western Electro-Chemical Co., U.S.P., 1,753,787, (*Chem. Abstracts*, 1930, **24**, 2471).

**Xanthone (Dibenz- $\gamma$ -pyrone)**

MW, 196

Needles from  $\text{EtOH}$ . M.p.  $174^\circ$ . B.p.  $349-50^\circ/730$  mm. Sol.  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ . Less sol.  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ . Spar. sol. ligroin. Insol. cold  $\text{H}_2\text{O}$ . Spar. volatile in steam. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with intense light blue fluor. Zn dust +  $\text{NaOH} \rightarrow$  xanthidrol. Zn dust dist.  $\rightarrow$  xanthene.

Oxime: cryst. M.p.  $161^\circ$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with blue fluor.

Phenylhydrazone: golden-yellow needles from  $\text{EtOH}$ . M.p.  $152^\circ$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with green fluor.

Anil: golden-yellow cryst. from  $\text{EtOH}$ . M.p.  $134.5^\circ$ . Insol. alkalis. Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with green fluor.

Perkin, *Ber.*, 1883, **16**, 339.

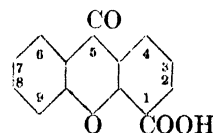
Graebe, *Ann.*, 1899, **254**, 280.

Graebe, Röder, *Ber.*, 1899, **32**, 1690.

Simon, *Bull. soc. chim. biol.*, 1926, **8**, 203.

Spektor, *Chem. Abstracts*, 1934, **28**, 575.

Holleman, *Organic Syntheses*, Collective Vol. I, 537.

**Xanthone-1-carboxylic Acid**

MW, 240

Needles from  $\text{PhNO}_2$ . M.p.  $289^\circ$  ( $275^\circ$ ). Sol. boiling  $\text{AcOH}$ ,  $\text{PhNO}_2$ , anisole, phenetole. Mod. sol. boiling  $\text{EtOH}$ . Insol.  $\text{CHCl}_3$ ,  $\text{CCl}_4$ ,  $\text{C}_6\text{H}_6$ . Cold conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellowish-green sol. Distills undecomp. Sublimes in needles.  $\text{KOH}$  fusion  $\rightarrow$  salicylic acid.

$\text{Me}$  ester:  $\text{C}_{15}\text{H}_{10}\text{O}_4$ . MW, 254. Yellowish needles from  $\text{MeOH}$ . M.p.  $146.5^\circ$ . Sol.  $\text{MeOH}$ . Insol.  $\text{H}_2\text{O}$ .

$\text{Et}$  ester:  $\text{C}_{16}\text{H}_{12}\text{O}_4$ . MW, 268. Yellowish microneedles from  $\text{EtOH}$ . M.p.  $123^\circ$ .

Chloride:  $\text{C}_{14}\text{H}_7\text{O}_3\text{Cl}$ . MW, 258.5. Cryst. from *sym.*-tetrachloroethane. M.p.  $165^\circ$ .

Amide:  $\text{C}_{14}\text{H}_9\text{O}_3\text{N}$ . MW, 239. Powder. M.p.  $320^\circ$ .

Anilide: needles from  $\text{EtOH}$ . M.p.  $252^\circ$ .

Perkin, *J. Chem. Soc.*, 1883, **43**, 188.

Anschütz, Claasen, *Ber.*, 1922, **55**, 686.

**Xanthone-3-carboxylic Acid.**

Cryst. from  $\text{AcOH}$  or  $\text{PhNO}_2$ . M.p.  $305^\circ$ .

$\text{Me}$  ester: needles from  $\text{MeOH}$ . M.p.  $185^\circ$ .

$\text{Et}$  ester: needles from  $\text{EtOH}$ . M.p.  $152^\circ$ .

Chloride: pale yellowish needles from  $\text{CHCl}_3$ . M.p.  $173^\circ$ .

Amide: powder. M.p.  $324^\circ$  decomp.

Anilide: needles from  $\text{EtOH}$ . M.p.  $271^\circ$ .

Anschütz, Stoltenhoff, Voeller, *Ber.*, 1925, **58**, 1740.

**Xanthophyll.**

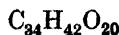
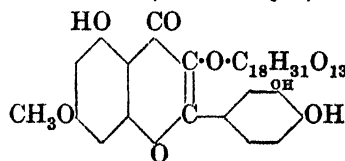
This name is now employed as a generic term for hydroxylated carotenoids, e.g. lutein, zeaxanthin, etc.

**Xanthopurpurin.**

See Purpuroxanthin.

**Xanthoquininic Acid.**

See 6-Hydroxycinchoninic Acid.

**Xanthorhamnin ( $\alpha$ -Rhamnegin)**

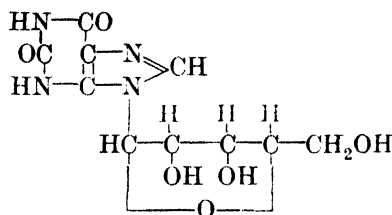
MW, 770

Yellow glucoside from Persian berries, *Rhamnus tinctoria*. Needles from EtOH-Et<sub>2</sub>O.  $[\alpha]_D + 3.75^\circ$ . Sol. EtOH. Insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Decomp. by H<sub>2</sub>O. FeCl<sub>3</sub> → dark brown col. Hyd. → rhamnetin + rhamnose.

Tri-Me ether: C<sub>37</sub>H<sub>48</sub>O<sub>20</sub>. MW, 812. Needles + 3H<sub>2</sub>O from H<sub>2</sub>O. M.p. 175–8°. Sol. H<sub>2</sub>O. FeCl<sub>3</sub> → brown col.

Attree, Perkin, *J. Chem. Soc.*, 1927, 238.  
Tanret, Tanret, *Bull. soc. chim.*, 1899, 21, 1073.

### Xanthosine (Xanthine 9-ribofuranoside)



Probable structure

C<sub>10</sub>H<sub>12</sub>O<sub>6</sub>N<sub>4</sub>

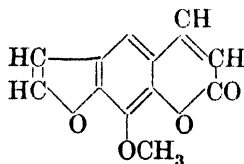
MW, 284

A typical nucleoside. Prismatic rods + 2H<sub>2</sub>O from H<sub>2</sub>O. Decomp. on heating without melting. Sol. hot H<sub>2</sub>O, EtOH. Aq. Spar. sol. cold H<sub>2</sub>O. Hyd. by min. acids → xanthine + ribose.

Phosphoric ester: see Xanthylic Acid.

Levene, Jacobs, *Ber.*, 1910, 43, 3163.  
Gulland, Macrae, *J. Chem. Soc.*, 1933, 667.  
Gulland, Holiday, Macrae, *J. Chem. Soc.*, 1934, 1640.

### Xanthotoxin (Zanthotoxin)



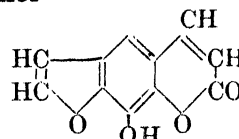
C<sub>12</sub>H<sub>8</sub>O<sub>4</sub>

MW, 216

Constituent of fruit of *Fagara xanthoxyloides*, Linn. Prisms from 80% EtOH, needles from C<sub>6</sub>H<sub>6</sub>-pet. ether. M.p. 146° (144–5°). Very sol. boiling EtOH. Mod. sol. Me<sub>2</sub>CO, AcOH. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O, pet. ether. Spar. volatile in steam. Cold conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. → brown on warming. Poisonous to fish.

Thoms, *Ber.*, 1911, 44, 3325.  
Späth, Pailer, *Ber.*, 1936, 69, 767.

### Xanthotoxol



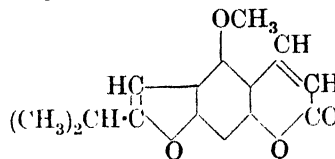
C<sub>11</sub>H<sub>6</sub>O<sub>4</sub>

MW, 202

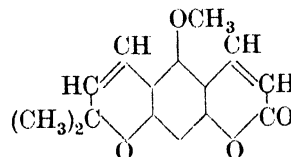
Present in seeds of *Angelica archangelica*. M.p. 251–2° (in evacuated tube). Diazo-methane → xanthotoxin.

Späth, Vierhapper, *Ber.*, 1937, 70, 248.

### Xanthoxyletin (Xanthoxylin-N)



or



C<sub>15</sub>H<sub>14</sub>O<sub>4</sub>

MW, 258

Constituent of bark of *Zanthoxylum americanum*, M. Prisms from pet. ether. M.p. 133°. Very sol. hot EtOH, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Less sol. Me<sub>2</sub>CO. Spar. sol. Et<sub>2</sub>O. Sol. 49 parts cold EtOH, 25,000 parts cold H<sub>2</sub>O. Cold conc. H<sub>2</sub>SO<sub>4</sub> → red sol. Tasteless.

Dieterle, Kruta, *Chem. Zentr.*, 1937, I, 4243.

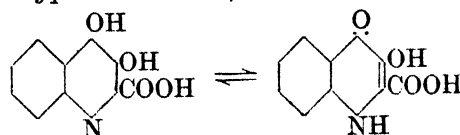
Gordin, *J. Am. Chem. Soc.*, 1906, 28, 1650.

Bell, Robertson, Subramaniam, *J. Chem. Soc.*, 1936, 627.

### Xanthoxylin-N.

See Xanthoxyletin.

**Xanthurenic Acid (Xanthuric acid, 3:4-dihydroxyquinoline-2-carboxylic acid, 3:4-dihydroxyquinaldinic acid)**



Probable structure

C<sub>10</sub>H<sub>7</sub>O<sub>4</sub>N

MW, 205

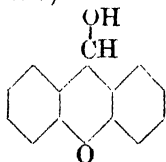
Yellow micro-cryst. from H<sub>2</sub>O. M.p. 286°. Sol. HCl, EtOH, AcOH + HCl. Spar. sol.

usual solvents. Caustic alkalis and carbonates  $\rightarrow$  yellow sols. Millon's reagent  $\rightarrow$  red col.  $\text{FeSO}_4 \rightarrow$  intense green col.

*Me ester*:  $\text{C}_{11}\text{H}_9\text{O}_4\text{N}$ . MW, 219. Yellow cryst. from MeOH. M.p.  $262^\circ$ . *Dibenzoyl*: m.p.  $171^\circ$ .

Musajo, *Chem. Zentr.*, 1935, II, 2079; 1936, II, 3540.

**Xanthydrol** (*Dibenz- $\gamma$ -pyranol*, 5-hydroxy-xanthenol, xanthenol-5)



$\text{C}_{13}\text{H}_{10}\text{O}_2$  MW, 198

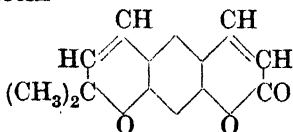
Needles from hot EtOH- $\text{H}_2\text{O}$ . M.p. about  $123^\circ$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  yellow sol. with green fluor. Warm in air  $\rightarrow$  xanthone. Forms salts with min. acids. Reagent for urea.

Kirkhof, Spektor, *Chem. Abstracts*, 1934, 28, 5451.

Kyn-Jones, Ward, *J. Chem. Soc.*, 1930, 535.

Holleman, *Organic Syntheses*, Collective Vol. I, 539.

### Xanthyletin

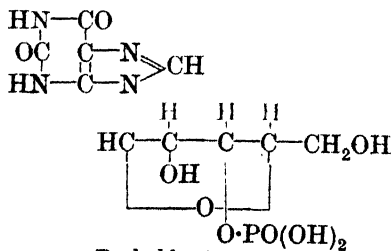


$\text{C}_{14}\text{H}_{12}\text{O}_3$  MW, 228

Constituent of bark of *Zanthoxylum americanum*, M. Prisms from petrol. M.p.  $128-128.5^\circ$ . Conc.  $\text{H}_2\text{SO}_4 \rightarrow$  orange-red sol. NaOH  $\rightarrow$  acetone + resorcinol.

Bell, Robertson, *J. Chem. Soc.*, 1936, 1828.

**Xanthylic Acid** (*Xanthine 9-ribosephosphoric acid*)



Probable structure

$\text{C}_{10}\text{H}_{15}\text{O}_9\text{N}_4\text{P}$  MW, 366

A typical nucleic acid. Powder from  $\text{H}_2\text{O}$ .  $[\alpha]_D^{20} - 61.66^\circ$  in 5% NaOH. Aq. sol. at  $50^\circ$

decomp. with formation of *d*-ribose-phosphoric acid.

*Brucine salt*: needles from 30% EtOH. M.p.  $200^\circ$ .

Knopf, *Z. physiol. Chem.*, 1914, 92, 160.

Levene, Dmochowski, *J. Biol. Chem.*, 1931, 93, 565.

Levene, Harris, *J. Biol. Chem.*, 1932, 95, 757.

### Xaxaquin.

See under Quinine.

### Xenylamine.

See Aminodiphenyl.

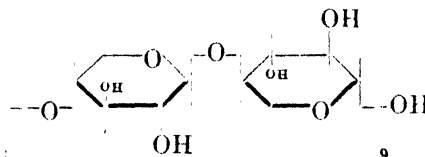
### Xenylcarbinol.

See Hydroxymethyldiphenyl.

### p-Xenyl Mercaptan.

See 4-Mercaptodiphenyl.

### Xylan



Arabo-  
furanose  
unit

Probable structure

$(\text{C}_5\text{H}_8\text{O}_4)_n$  MW, (132)<sub>n</sub>

Occurs in wood gum, straw, esparto grass, maize cobs, oat hulls, hemp stalk, and is one of the hemicelluloses present in wood. White powder. M.p.  $198^\circ$ .  $[\alpha]_D^{20} - 109.5^\circ$  in 2.5% aq. NaOH (for xylan containing 10%  $\text{H}_2\text{O}$ ). Does not reduce Fehling's. Hyd. by 3%  $\text{HNO}_3 \rightarrow$  xylose (93%) + arabinose.

"Dimethyl ether": m.p.  $194-6^\circ$  ( $198^\circ$ ). B.p. about  $80^\circ/0.04$  mm.  $n_D^{17} 1.4581$ .  $[\alpha]_D^{20} + 61.8^\circ$  in MeOH,  $[\alpha]_D^{20} - 98.3^\circ$  in  $\text{CHCl}_3$ . Sol. AcOH,  $\text{Me}_2\text{CO}$ ,  $\text{CHCl}_3$ . Spar. sol.  $\text{Et}_2\text{O}$ .

"Diacetyl": amorph. powder. Sol. Py. Spar. sol.  $\text{CHCl}_3$ . Insol.  $\text{H}_2\text{O}$ , MeOH, EtOH,  $\text{Et}_2\text{O}$ .

Salkowski, *Z. physiol. Chem.*, 1901, 34, 162.

Hurd, Currie, *J. Am. Chem. Soc.*, 1933, 55, 1521.

Heuser, *J. prakt. Chem.*, 1921, 103, 69.

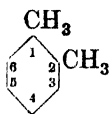
Heuser, Schlosser, *Ber.*, 1923, 56, 392.

Hibino, *J. Chem. Soc. Japan*, 1930, 51, 417.

Hampton, Haworth, Hirst, *J. Chem. Soc.*, 1929, 1739.

Haworth, Percival, *J. Chem. Soc.*, 1931, 2850.

Haworth, Hirst, Oliver, *J. Chem. Soc.*, 1934, 1917.

**o-Xylene** (1 : 2-Dimethylbenzene) $C_8H_{10}$ 

MW, 106

Colourless mobile liq. M.p.  $-25.0^\circ$ . B.p.  $143.95-144.15^\circ$  ( $142.3^\circ$ ). Vap. press.: 4.5 mm. at  $0^\circ$ , 10 mm. at  $20^\circ$ , 34.5 mm. at  $50^\circ$ , 199.5 mm. at  $100^\circ$ .  $D_4^{20}$  0.89679.  $n_D^{16}$  1.50712. Sol. EtOH, Et<sub>2</sub>O.  $CrO_2Cl_2 \rightarrow$  o-toluic aldehyde.  $KMnO_4 \rightarrow$  phthalic acid + o-toluic acid.  $CO + HCl$  in presence of  $AlCl_3 + Cu_2Cl_2 \rightarrow$  3 : 4-dimethylbenzaldehyde.

*Picrate*: lemon-yellow cryst. from EtOH. M.p.  $88.5^\circ$ .

I.G., D.R.P., 567,331, (*Chem. Abstracts*, 1933, 27, 1366); F.P., 639,252, (*Chem. Abstracts*, 1929, 23, 611).

General Aniline Works, U.S.P., 1,727,682, (*Chem. Abstracts*, 1929, 23, 5196).

**m-Xylene** (1 : 3-Dimethylbenzene).

Colourless, mobile liq. M.p.  $-47.4^\circ$ . B.p.  $139.30^\circ$ . Vap. press.: 2 mm. at  $0^\circ$ , 6 mm. at  $20^\circ$ , 31 mm. at  $50^\circ$ , 218 mm. at  $100^\circ$ .  $D_4^{20}$  0.88113.  $D_4^{25}$  0.86835.  $n_D^{15}$  1.49989. Sol. EtOH, Et<sub>2</sub>O.  $CrO_3 \rightarrow$  isophthalic acid.

*Picrate*: lemon-yellow cryst. from EtOH. M.p.  $90-91.5^\circ$ .

I.G., D.R.P., 567,331, (*Chem. Abstracts*, 1933, 27, 1366).

**p-Xylene** (1 : 4-Dimethylbenzene).

Plates or prisms. M.p.  $13-14^\circ$ . B.p.  $137.5-138.0^\circ$ . Vap. press.: 2 mm. at  $0^\circ$ , 6 mm. at  $20^\circ$ , 32 mm. at  $50^\circ$ , 230 mm. at  $100^\circ$ .  $D_4^{20}$  0.8541.  $n_D^{21}$  1.5004. Sol. EtOH, Et<sub>2</sub>O. Dil.  $HNO_3 \rightarrow$  p-toluic acid.  $CrO_3 \rightarrow$  terephthalic acid.

*Picrate*: lemon-yellow cryst. from EtOH. M.p.  $90.5^\circ$ .

Jannasch, *Ber.*, 1877, 10, 1356.

I.G., D.R.P., 567,331, (*Chem. Abstracts*, 1933, 27, 1366); F.P., 639,252, (*Chem. Abstracts*, 1929, 23, 611).

General Aniline Works, U.S.P., 1,727,682, (*Chem. Abstracts*, 1929, 23, 5196).

**Xyleneazocresol.**

See 6-Hydroxy-3 : 2' : 4'-trimethylazobenzene and 6-Hydroxy-3 : 3' : 4'-trimethylazobenzene.

**Xyleneazophenetole.**

See under 4'-Hydroxy-2 : 4-dimethylazobenzene.

**Xyleneazophenol.**

See 4'-Hydroxy-2 : 4-dimethylazobenzene.

**m-Xyleneazo-m-5-xenol.**

See 4-Hydroxy-2 : 6 : 2' : 4'-tetramethylazobenzene.

**Xyleneazoxylidine.**

See Aminotetramethylazobenzene.

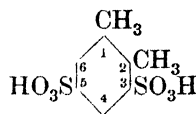
**Xylene-carboxylic Acid.**

See Dimethylbenzoic Acid.

**Xylene-dicarboxylic Acid.**

See Dimethylphthalic Acid, Dimethylisophthalic Acid, Dimethylterephthalic Acid, and Phenylenediacetic Acid.

**o-Xylene-3 : 5-disulphonic Acid** (4 : 5-Dimethylbenzene-1 : 3-disulphonic acid)

 $C_8H_{10}O_6S_2$ 

MW, 266

*Dichloride*:  $C_8H_8O_4Cl_2S_2$ . MW, 303. Yellow prisms from Et<sub>2</sub>O. M.p.  $79^\circ$ . Sol. Et<sub>2</sub>O,  $CHCl_3$ ,  $CS_2$ .

*Diamide*:  $C_8H_{12}O_4N_2S_2$ . MW, 264. Cryst. M.p.  $239^\circ$ . Mod. sol. H<sub>2</sub>O.

*Dianilide*: cryst. from EtOH. M.p.  $199-200^\circ$ . Very sol. Me<sub>2</sub>CO. Sol. EtOH. Spar. sol. H<sub>2</sub>O.

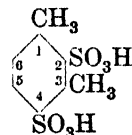
Pollak, Heimberg-Krauss, Katscher, Lustig, *Monatsh.*, 1930, 55, 365.

**o-Xylene-3 : 6-disulphonic Acid** (2 : 3-Dimethylbenzene-1 : 4-disulphonic acid).

*Diamide*: cryst. from EtOH.Aq. M.p.  $251^\circ$ .

Holleman, Choufoer, *Chem. Abstracts*, 1924, 18, 3183.

**m-Xylene-2 : 4-disulphonic Acid** (2 : 4-Dimethylbenzene-1 : 3-disulphonic acid)

 $C_8H_{10}O_6S_2$ 

MW, 266

*Dichloride*:  $C_8H_8O_4Cl_2S_2$ . MW, 303. Viscous oil.

*Diamide*:  $C_8H_{12}O_4N_2S_2$ . MW, 264. Needles from H<sub>2</sub>O. M.p.  $223-4^\circ$  ( $235^\circ$ ).

Pollak, v. Meissner, *Monatsh.*, 1928, 50, 247.

Choufoer, *Chem. Abstracts*, 1925, 19, 2195.



**m-Xylene-4 : 6-disulphonic Acid** (4 : 6-Dimethylbenzene-1 : 3-disulphonic acid).

Needles. Decomp. very readily.

*Di-Et ester*:  $C_{12}H_{18}O_6S_2$ . MW, 322. Leaflets. Sol. EtOH. Insol.  $H_2O$ .

*Diffuoride*:  $C_8H_8O_4F_2S_2$ . MW, 270. Cryst. M.p. 116–18°.

*Dichloride*: cryst. from pet. ether. M.p. 130°. Mod. sol.  $Et_2O$ ,  $CHCl_3$ ,  $CS_2$ .

*Diamide*: needles from  $H_2O$ . M.p. 249°. Spar. sol.  $H_2O$ . N : N'-*Di-Et*: needles from  $H_2O$ . M.p. 135°.

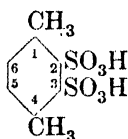
*Dianilide*: cryst. from 50% EtOH. M.p. 196°.

Pollak, v. Meissner, *Monatsh.*, 1928, 50, 244.

Davies, Dick, *J. Chem. Soc.*, 1931, 2107.

Holleman, Choufoer, *Rec. trav. chim.*, 1929, 48, 1076.

**p-Xylene-2 : 3-disulphonic Acid** (3 : 6-Dimethylbenzene-1 : 2-disulphonic acid)



$C_8H_{10}O_6S_2$  MW, 266

*Anhydride*:  $C_8H_8O_5S_2$ . MW, 248. Leaflets from pet. ether. M.p. 189–90°.

Holleman, Choufoer, *Rec. trav. chim.*, 1929, 48, 1082.

**p-Xylene-2 : 6-disulphonic Acid** (2 : 5-Dimethylbenzene-1 : 3-disulphonic acid).

Needles. Very sol.  $H_2O$ .

*Dichloride*:  $C_8H_8O_4Cl_2S_2$ . MW, 303. Cryst. from ligroin. M.p. 81°. Sol.  $Et_2O$ , AcOEt,  $CHCl_3$ ,  $C_6H_6$ . Spar. sol.  $CS_2$ , pet. ether, ligroin.

*Diamide*:  $C_8H_{12}O_4N_2S_2$ . MW, 264. Cryst. from EtOH.Aq. M.p. 294–5° decomp. Sol.  $Me_2CO$ . Spar. sol.  $Et_2O$ , AcOH,  $CHCl_3$ . Very spar. sol.  $H_2O$ .

*Dianilide*: cryst. from EtOH. M.p. 174°.

Holmes, *Am. Chem. J.*, 1891, 13, 372.

Pollak, Schadler, *Monatsh.*, 1918, 39, 144.

Pollak, Heimberg-Krauss, Katscher, Lustig, *Monatsh.*, 1930, 55, 366.

**p-Xylene-3 : 6-disulphonic Acid** (2 : 5-Dimethylbenzene-1 : 4-disulphonic acid).

*Dichloride*: cryst. from pet. ether. M.p. 164°.

*Diamide*: m.p. about 310° (297°).

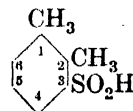
*Dianilide*: cryst. from EtOH. M.p. 223°.

See last reference above.

**Xylene Musk.**

See 2 : 4 : 6-Trinitro-5-*tert.*-butyl-*m*-xylene.

**o-Xylene-3-sulphinic Acid** (2 : 3-Dimethylbenzenesulphinic acid)



$C_8H_{10}O_2S$

MW, 170

Cryst. M.p. 105°. Sol.  $Et_2O$ .

Moschner, *Ber.*, 1901, 34, 1260.

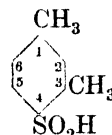
**o-Xylene-4-sulphinic Acid** (3 : 4-Dimethylbenzenesulphinic acid).

Plates from  $H_2O$ . M.p. 83°.

Jacobsen, *Ber.*, 1877, 10, 1010.

Knoevenagel, Kenner, *Ber.*, 1908, 41, 3319.

**m-Xylene-4-sulphinic Acid** (2 : 4-Dimethylbenzenesulphinic acid)



$C_8H_{10}O_2S$

MW, 170

Needles from  $H_2O$ . M.p. 77–8°.

Gattermann, *Ber.*, 1899, 32, 1141.

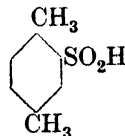
Knoevenagel, Kenner, *Ber.*, 1908, 41, 3318.

**m-Xylene-5-sulphinic Acid** (3 : 5-Dimethylbenzenesulphinic acid).

Reddish cryst. mass. M.p. 75–6°.

Moschner, *Ber.*, 1901, 34, 1260.

**p-Xylene-2-sulphinic Acid** (2 : 5-Dimethylbenzenesulphinic acid)



$C_8H_{10}O_2S$

MW, 170

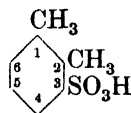
Needles from  $H_2O$ . M.p. 85°. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

*Anhydride*: m.p. 68–9°.

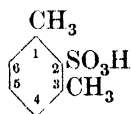
Hilditch, *J. Chem. Soc.*, 1908, 93, 1527.

Gattermann, *Ber.*, 1899, 32, 1141.

Knoevenagel, Polack, *Ber.*, 1908, 41, 3327.

**o-Xylene-3-sulphonic Acid** (2:3-Dimethylbenzenesulphonic acid) $C_8H_{10}O_3S$ 

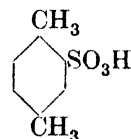
MW, 186

Cryst. from  $H_2O$ . Heat in air at  $115-20^\circ \rightarrow$  4-sulphonic acid.**Chloride**:  $C_8H_9O_2ClS$ . MW, 204.5. Prisms from pet. ether. M.p.  $47^\circ$ .**Amide**:  $C_8H_{11}O_2NS$ . MW, 185. Needles from  $H_2O$ . M.p.  $167^\circ$  ( $165^\circ$ ). Spar. sol.  $H_2O$ .Moody, *Chem. News*, 1893, **67**, 34.Moschner, *Ber.*, 1901, **34**, 1260.**o-Xylene-4-sulphonic Acid** (3:4-Dimethylbenzenesulphonic acid).Plates or prisms +  $2H_2O$  from  $CHCl_3$ . M.p.  $63-4^\circ$  ( $55^\circ$ ). Very hygroscopic.**Chloride**: prisms from  $Et_2O$ . M.p.  $51-2^\circ$ .**Amide**: prisms from  $EtOH$ . M.p.  $143-4^\circ$ .Patterson, McMillan, Somerville, *J. Chem. Soc.*, 1924, **125**, 2489.Jacobsen, *Ber.*, 1877, **10**, 1010.**m-Xylene-2-sulphonic Acid** (2:6-Dimethylbenzenesulphonic acid) $C_8H_{10}O_3S$ 

MW, 186

Cryst. Heat at  $100^\circ \rightarrow$  4-sulphonic acid.**Chloride**:  $C_8H_9O_2ClS$ . MW, 204.5. Prisms. M.p.  $39^\circ$ .**Amide**:  $C_8H_{11}O_2NS$ . MW, 185. Needles. M.p.  $113^\circ$ .Pollak, v. Meissner, *Monatsh.*, 1928, **50**, 246.Moody, *Chem. News*, 1888, **58**, 21.**m-Xylene-4-sulphonic Acid** (2:4-Dimethylbenzenesulphonic acid).Plates or prisms +  $2H_2O$  from  $H_2O$ . M.p.  $61-2^\circ$ .**Fluoride**:  $C_8H_9O_2FS$ . MW, 188. B.p.  $246^\circ$  ( $239-40^\circ$ ),  $149-50^\circ/15$  mm.  $n_D^{20}$  1.5086.**Chloride**: cryst. M.p.  $34^\circ$ .**Amide**: needles from  $H_2O$ . M.p.  $138-9^\circ$  ( $137^\circ$ ). N-Benzoyl: needles from  $EtOH$ . M.p.  $149-51^\circ$ .**Methylamide**:  $C_9H_{13}O_2NS$ . MW, 199. Cryst. from  $EtOH$ . M.p.  $43^\circ$ . Sol.  $EtOH$ ,  $Et_2O$ .

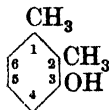
Dict. of Org. Comp.—III.

**Dimethylamide**:  $C_{10}H_{15}O_2NS$ . MW, 213. Cryst. from  $EtOH.Aq$ . M.p.  $35^\circ$ .**Anilide**: cryst. from  $EtOH$ . M.p.  $109-10^\circ$ . N-Me: m.p.  $55^\circ$ .**p-Nitroanilide**: cryst. +  $C_6H_6$  from  $C_6H_6$ . M.p.  $91-3^\circ$ , solvent free  $117-19^\circ$ .**Anhydride**: m.p.  $139^\circ$ . Decomp. on standing.Crafts, *Ber.*, 1901, **34**, 1352.Pollak, v. Meissner, *Monatsh.*, 1928, **50**, 246.Davies, Dick, *J. Chem. Soc.*, 1931, 2107.Patterson, McMillan, Somerville, *J. Chem. Soc.*, 1924, **125**, 2489.**m-Xylene-5-sulphonic Acid** (3:5-Dimethylbenzenesulphonic acid).Needles from  $H_2O$ .**Chloride**: needles from pet. ether or  $C_6H_6$ . M.p.  $94^\circ$  ( $89-90^\circ$ ).**Bromide**:  $C_8H_9O_2BrS$ . MW, 249. M.p.  $92-3^\circ$ .**Amide**: prisms from boiling  $EtOH$ . M.p.  $135^\circ$ . Sol.  $EtOH$ . Less sol.  $Et_2O$ . Mod. sol. hot  $H_2O$ . Insol. ligroin.**Anilide**: cryst. from  $EtOH$ . M.p.  $119^\circ$ .**p-Toluidide**: prisms from  $EtOH$ . M.p.  $121-2^\circ$ .Moschner, *Ber.*, 1901, **34**, 1260.Jungahn, *Ber.*, 1902, **35**, 3756.Armstrong, Wilson, *Chem. News*, 1901, **83**, 46.**p-Xylene-2-sulphonic Acid** (2:5-Dimethylbenzenesulphonic acid) $C_8H_{10}O_3S$ 

MW, 186

Leaflets or prisms +  $2H_2O$  from  $H_2O$ . M.p.  $86^\circ$ . Sol.  $CHCl_3$ .**Fluoride**:  $C_8H_9O_2FS$ . MW, 188. M.p.  $24.5^\circ$ . B.p.  $124-5^\circ/21$  mm.**Chloride**:  $C_8H_9O_2ClS$ . MW, 204.5. Prisms. M.p.  $24-6^\circ$ .**Amide**:  $C_8H_{11}O_2NS$ . MW, 185. Needles. M.p.  $147-8^\circ$ . Sol.  $EtOH$ . Mod. sol. hot  $H_2O$ .Jacobsen, *Ber.*, 1877, **10**, 1009; 1878, **11**, 22.Steinkopf et al., *J. prakt. Chem.*, 1927, **117**, 39.

**o-3-Xylenol** (2:3-Dimethylphenol, 3-hydroxy-o-xylene)



$C_8H_{10}O$  MW, 122

Needles from EtOH.Aq. M.p. 75° (73.5°). B.p. 218°.  $FeCl_3 \rightarrow$  blue col.

*Me ether*: 2:3-dimethylanisole.  $C_9H_{12}O$ . MW, 136. Cryst. M.p. 29°. B.p. 199°. Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ .

*Et ether*: 2:3-dimethylphenetole.  $C_{10}H_{14}O$ . MW, 150. M.p. 10°. B.p. 212.5°.

Moschner, *Ber.*, 1900, **33**, 742.

Short, Stromberg, Wiles, *J. Chem. Soc.*, 1936, 322.

Kruber, Schmitt, *Ber.*, 1931, **64**, 2270.

**o-4-Xylenol** (3:4-Dimethylphenol, 4-hydroxy-o-xylene).

Needles from  $H_2O$ . M.p. 62.5°. B.p. 225°/757 mm. Heat of comb.  $C_p$  1035.4 Cal.  $k = 5.2 \times 10^{-11}$  at 25°.

*Me ether*: 3:4-dimethylanisole. B.p. 204–5°.

*Et ether*: 3:4-dimethylphenetole. B.p. 218°.

3:5-Dinitrobenzoyl: needles from 95% EtOH. M.p. 181.6°.

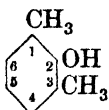
1-Naphthylurethane: m.p. 141–2°.

Picrate: chrome yellow. M.p. 83.8°.

Jacobsen, *Ber.*, 1878, **11**, 28; 1884, **17**, 161.

Moschner, *Ber.*, 1900, **33**, 743.

**m-2-Xylenol** (2:6-Dimethylphenol, 2-hydroxy-m-xylene)



$C_8H_{10}O$  MW, 122

Leaflets or flat needles. M.p. 49°. B.p. 203°.

*Me ether*: 2:6-dimethylanisole.  $C_9H_{12}O$ . MW, 136. B.p. 182–3°.

*Et ether*: 2:6-dimethylphenetole.  $C_{10}H_{14}O$ . MW, 150. B.p. 195.5–196.5°.  $D_4^{13.9}$  0.9420.  $n_D^{25.9}$  1.497.

3:5-Dinitrobenzoyl: plates from 95% EtOH. M.p. 158.8°.

Picrate: orange-yellow. M.p. 50–3°.

Bamberger, *Ber.*, 1903, **36**, 2036.

Gattermann, *Ann.*, 1907, **357**, 363.

**m-4-Xylenol** (2:4-Dimethylphenol, 4-hydroxy-m-xylene).

Needles. M.p. 27–8°. B.p. 211.5°/766 mm., 97–8°/14 mm.  $D_4^{14}$  1.0276.  $n_D^{14}$  1.5420. Misc.

with EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ . Heat of comb.  $C_p$  1037.5 Cal.

*Me ether*: 2:4-dimethylanisole. B.p. 192°.  $D_4^{13.45}$  0.9691.  $n_D^{13.55}$  1.517.

*Et ether*: 2:4-dimethylphenetole. B.p. 202–3°.  $D_4^{13.9}$  0.9488.  $n_D^{13.95}$  1.507.

*Acetyl*: b.p. 107.5–108.5°/13 mm.  $D_4^{15.5}$  1.0298.  $n_D^{15.5}$  1.4990.

*Propionyl*: b.p. 121.5°/15 mm.  $D_4^{17}$  1.0104.  $n_D^{17}$  1.4944.

*Benzoyl*: cryst. from AcOH. M.p. 37–8°. B.p. 110.5–111°/15 mm.

3:5-Dinitrobenzoyl: plates from 95% EtOH. M.p. 164.6°.

Phenylurethane: m.p. 102°.

1-Naphthylurethane: m.p. 134–5°.

Sabatier, Mailhe, *Compt. rend.*, 1910, **151**, 361.

Palfray, Duboc, *Compt. rend.*, 1927, **185**, 1479.

**m-5-Xylenol** (3:5-Dimethylphenol, 5-hydroxy-m-xylene).

Needles from  $H_2O$ . M.p. 68° (64°). B.p. 219.5°. Sublimes.

*Me ether*: 3:5-dimethylanisole. B.p. 193°.

*Et ether*: 3:5-dimethylphenetole. B.p. 208°.

*Isopropyl ether*:  $C_{11}H_{16}O$ . MW, 164. B.p. 208–10°.

*Allyl ether*:  $C_{11}H_{14}O$ . MW, 162. B.p. 109°/11 mm.

*Acetyl*: b.p. 130°/26 mm., 120°/11 mm.

*Chloroacetyl*: b.p. 175–7°/48 mm., 146–7°/12 mm.

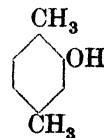
3:5-Dinitrobenzoyl: rods from 95% EtOH. M.p. 195.4°.

Phenylurethane: m.p. 151°.

Raschig, D.R.P., 254,716, (*Chem. Zentr.*, 1913, I, 353).

Auwers, Borsche, *Ber.*, 1915, **48**, 1708, 1722.

**p-2-Xylenol** (2:5-Dimethylphenol, hydroxy-p-xylene)



$C_8H_{10}O$  MW, 122

Prisms from EtOH– $Et_2O$ . M.p. 75°. B.p. 211.5°/762 mm. Sol. EtOH,  $Et_2O$ . Heat of comb.  $C_p$  1035.6 Cal.  $k = 4.8 \times 10^{-11}$  at 25°. No col. with  $FeCl_3$ . Sublimes. Volatile in steam.

*Me ether*: 2:5-dimethylanisole.  $C_9H_{12}O$ . MW, 136. B.p. 194°/772 mm.

*Et ether*: 2:5-dimethylphenetole.  $C_{10}H_{14}O$ . MW, 150. B.p.  $198.8^{\circ}/748$  mm. Heat of comb.  $C_p$  1368.85 Cal.

*Acetyl*: b.p.  $237^{\circ}/768$  mm.

3:5-Dinitrobenzoyl: needles from 95% EtOH. M.p.  $137.2^{\circ}$ .

*Phenylurethane*: m.p.  $162^{\circ}$  ( $160-1^{\circ}$ ).

1-Naphthylurethane: m.p.  $172-3^{\circ}$ .

*Picrate*: orange. M.p.  $81-2^{\circ}$ .

Morgan, Pettet, E.P., 397,148, (*Chem. Abstracts*, 1934, 28, 784).

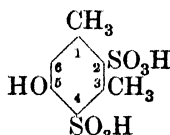
Jacobsen, *Ber.*, 1878, 11, 26.

Noelting, Witt, Forel, *Ber.*, 1885, 18, 2665.

### Xylenol-carboxylic Acid.

See Hydroxydimethylbenzoic Acid.

**m-5-Xylenol-2:4-disulphonic Acid** (3:5-Dimethylphenol-2:4-disulphonic acid, 6-hydroxy-2:4-dimethylbenzene-1:3-disulphonic acid)



$C_8H_{10}O_7S_2$  MW, 282

*Dichloride*:  $C_8H_8O_5Cl_2S_2$ . MW, 319. M.p.  $117-19^{\circ}$ .

*Diamide*:  $C_8H_{12}O_5N_2S_2$ . MW, 280. Leaflets from  $H_2O$ . M.p.  $206-8^{\circ}$ .

*Dianilide*: cryst. from  $C_6H_6$ . M.p.  $205-7^{\circ}$ .

Katscher, Lehr, *Monatsh.*, 1934, 64, 238.

Ler, *Chem. Abstracts*, 1934, 28, 4714.

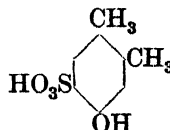
**m-5-Xylenol-4:6-disulphonic Acid** (3:5-Dimethylphenol-2:6-disulphonic acid, 2-hydroxy-4:6-dimethylbenzene-1:3-disulphonic acid).

*Dichloride*: cryst. from pet. ether. M.p.  $89-91^{\circ}$ .

*Dianilide*: m.p.  $160-1^{\circ}$ .

See last reference above.

**o-4-Xylenol-5-sulphonic Acid** (3:4-Dimethylphenol-6-sulphonic acid, 2-hydroxy-4:5-dimethylbenzenesulphonic acid)



$C_8H_{10}O_4S$  MW, 202

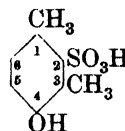
*Cryst.* Steam at  $107-10^{\circ} \longrightarrow$  o-4-xylenol.

*Na salt*: prisms.

*Ba salt*: rhombic plates. Spar. sol. cold  $H_2O$ .

Jacobsen, *Ber.*, 1878, 11, 28.

**m-4-Xylenol-2-sulphonic Acid** (2:4-Dimethylphenol-3-sulphonic acid, 3-hydroxy-2:6-dimethylbenzenesulphonic acid)



$C_8H_{10}O_4S$

MW, 202

*Cryst.*

*Ba salt*: needles.

Jacobsen, *Ann.*, 1879, 195, 283.

**m-4-Xylenol-5-sulphonic Acid** (2:4-Dimethylphenol-6-sulphonic acid, 2-hydroxy-3:5-dimethylbenzenesulphonic acid).

Needles. Very sol.  $H_2O$ , EtOH.  $FeCl_3 \longrightarrow$  blue col.  $\longrightarrow$  green on adding EtOH. Decomp. with steam at  $121-5^{\circ}$ .

*Fluoride*:  $C_8H_9O_3FS$ . MW, 204. B.p.  $71-3^{\circ}$  in high vacuum.

*Chloride*:  $C_8H_9O_3ClS$ . MW, 220.5. *Acetyl*: cryst. from pet. ether. M.p.  $62^{\circ}$ .

*Anilide*: leaflets from EtOH.Aq. M.p.  $142-3^{\circ}$ .

*N-Me*: prisms from pet. ether. M.p.  $111-12^{\circ}$ .

*N-Acetyl*: cryst. from pet. ether. M.p.  $105^{\circ}$ .

Katscher, Lehr, *Monatsh.*, 1934, 64, 242.

Ler, *Chem. Abstracts*, 1934, 28, 4715.

**m-4-Xylenol-6-sulphonic Acid** (2:4-Dimethylphenol-5-sulphonic acid, 3-hydroxy-4:6-dimethylbenzenesulphonic acid).

Needles. Very sol.  $H_2O$ , EtOH.  $FeCl_3 \longrightarrow$  bluish-violet col.

*Me ether*:  $C_9H_{12}O_4S$ . MW, 216. Needles. Sol.  $H_2O$ , EtOH,  $Et_2O$ . Spar. sol.  $C_6H_6$ . Decomp. on heating. *Amide*:  $C_9H_{13}O_3NS$ . MW, 215. Needles. M.p.  $190^{\circ}$ . Sol. EtOH. Spar. sol.  $H_2O$ .

*Et ether*:  $C_{10}H_{14}O_4S$ . MW, 230. Plates. Sol. EtOH. *Chloride*:  $C_{10}H_{13}O_3ClS$ . MW, 248.5. Rhombic plates from ligroin. M.p.  $56^{\circ}$ . *Amide*:  $C_{10}H_{15}O_3NS$ . Needles from EtOH. M.p.  $169-70^{\circ}$ .

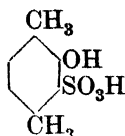
*Propyl ether*:  $C_{11}H_{16}O_4S$ . MW, 244. Plates or needles. Mod. sol.  $H_2O$ , EtOH. *Amide*:  $C_{11}H_{17}O_3NS$ . Needles. M.p.  $146^{\circ}$ . Sol. EtOH. Spar. sol.  $H_2O$ .

Sartig, *Ann.*, 1885, 230, 336.

Shober, Kiffer, *Am. Chem. J.*, 1897, 19, 386.

Moody, *Chem. News*, 1892, 65, 60.

**p-2-Xylenol-3-sulphonic Acid** (2:5-Dimethylphenol-6-sulphonic acid, 2-hydroxy-3:6-dimethylbenzenesulphonic acid)



$C_8H_{10}O_4S$

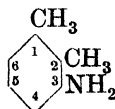
MW, 202

Cryst. from  $H_2O$ . Decomp. with steam at 115–18°.

*Ba salt*: needles.

Morgan, Pettet, E.P., 397,148, (*Chem. Abstracts*, 1934, 28, 784).  
Jacobsen, *Ber.*, 1878, 11, 27.

**o-3-Xylidine** (3-Amino-o-xylene)



$C_8H_{11}N$

MW, 121

Liq. B.p. 221–2°.  $D^{15}_D$  0.991.

*B, HCl*: m.p. 254°.

*N-Me*: see *N-Methyl-o-3-xylidine*.

*N-Di-Me*: see *N-Dimethyl-o-3-xylidine*.

*N-Et*:  $C_{10}H_{15}N$ . MW, 149. B.p. 227–8°.

*N-Acetyl*: 3-acet-1:2-xylidide. Needles from EtOH or  $H_2O$ . M.p. 135° (131°). Sol. EtOH, Et<sub>2</sub>O. Mod. sol.  $C_6H_6$ .

Hodgkinson, Limpach, *J. Chem. Soc.*, 1900, 77, 68.

Menton, *Ann.*, 1891, 263, 321.

**o-4-Xylidine** (4-Amino-o-xylene).

Plates or prisms from ligroin. M.p. 51° (47–8°). B.p. 226°. Mod. sol. pet. ether. Spar. sol. cold  $H_2O$ .

*B, HCl*: m.p. 256°.

*N-Formyl*: m.p. 52°.

*N-Di-Me*: see *N-Dimethyl-o-4-xylidine*.

*N-Acetyl*: 4-acet-1:2-xylidide. Prisms or needles from EtOH.Aq. M.p. 99°. Very sol. EtOH.

*N-Chloroacetyl*: needles from EtOH.Aq. M.p. 109°. Sol. EtOH, Et<sub>2</sub>O, AcOH.

*N-Cinnamoyl*: m.p. 175–6°.

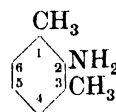
Bamberger, Blangey, *Ann.*, 1911, 384, 318, Note 2.

Jacobsen, *Ber.*, 1884, 17, 160.

Graebe, *Ber.*, 1901, 34, 1779.

Limpach, *Ber.*, 1888, 21, 646.

**m-2-Xylidine** (2-Amino-m-xylene)



$C_8H_{11}N$

MW, 121

Liq. B.p. 214°/739 mm. Ox. → *m*-xyloquinone.

*N-Me*: see *N-Methyl-m-2-xylidine*.

*N-Di-Me*: see *N-Dimethyl-m-2-xylidine*.

*N-Et*:  $C_{10}H_{15}N$ . MW, 149. Oil. B.p. 217–18°.

*N-Di-Et*:  $C_{12}H_{19}N$ . MW, 177. B.p. 220–21°. Volatile in steam.

*N-Formyl*: needles from EtOH. M.p. 176–7° (rapid heat.). Mod. sol. EtOH.

*N-Acetyl*: 2-acet-1:3-xylidide. Needles. M.p. 177°. *N-Nitroso*: plates from pet. ether. M.p. 62–3°. Heat in  $C_6H_6$  → 7-methylindazole.

*N-Benzoyl*: needles from EtOH.Aq. M.p. 168–168.5°. Very sol. EtOH, Et<sub>2</sub>O,  $CHCl_3$ . Spar. sol. cold  $C_6H_6$ , ligroin.

Friedlander, Brand, *Monatsh.*, 1898, 19, 639.

Busch, *Ber.*, 1899, 32, 1008.

I.C.I., U.S.P., 1,867,962, (*Chem. Abstracts*, 1932, 26, 5106); E.P., 328,418, (*Chem. Abstracts*, 1930, 24, 5309).

**m-4-Xylidine** (4-Amino-m-xylene).

B.p. 215.8–216.0°/728 mm.  $D^{19}_D$  0.9783.  $n^{19}_D$  1.56066. Heat of comb.  $C_e$  1111.42 Cal.  $k = 6.3 \times 10^{-10}$  at 15°.

*B, HCl*: prisms from  $H_2O$ . M.p. 235–6°.

*Oxalate*: m.p. 167°.

*Picrate*: yellow cryst. powder. M.p. 209° decomp.

*N-Me*: see *N-Methyl-m-4-xylidine*.

*N-Di-Me*: see *N-Dimethyl-m-4-xylidine*.

*N-Butyl*:  $C_{12}H_{19}N$ . MW, 177. Pale yellow oil. B.p. 267–70°/765 mm., 162–4°/29 mm. *Acetyl*: b.p. 290–3°/765 mm. *Benzoyl*: m.p. 193°.

*N-Formyl*: plates or needles from  $H_2O$ . M.p. 113–14°. Sol. EtOH, Et<sub>2</sub>O.

*N-Acetyl*: 4-acet-1:3-xylidide. Needles from EtOH.Aq. M.p. 129–30°. B.p. 170°/10 mm. Sol. EtOH. Spar. sol.  $H_2O$ .

*N-Chloroacetyl*: needles from  $C_6H_6$  or EtOH. M.p. 151–2°. Sol. EtOH. Spar. sol.  $C_6H_6$ .

*N-Benzoyl*: needles from EtOH. M.p. 192°. Sol. EtOH. Insol.  $H_2O$ .

*N-p-Nitrobenzoyl*: prisms from EtOH.Aq. M.p. 166°.

N-p-Toluenesulphonyl: m.p. 180.4–181.3°.

Pinnow, Oesterreich, *Ber.*, 1898, **31**, 2930.  
Hodgkinson, Limpach, *J. Chem. Soc.*,  
1900, **77**, 66.

Willgerodt, Schmierer, *Ber.*, 1905, **38**,  
1473.

Reilly, O'Neill, *J. Soc. Chem. Ind.*, 1927,  
**46**, 226r.

Silesia Verein, F.P., 691,911, (*Chem.*  
*Abstracts*, 1931, **25**, 1265).

**m-5-Xylidine** (5-Amino-m-xylene).

B.p. 220–1°.  $D_4^{20}$  0.9935.

B,HCl: needles. Sublimes.

N-Di-Me: see N-Dimethyl-m-5-xylidine.

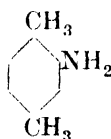
N-Formyl: prisms from EtOH.Aq. M.p.  
76.5°.

N-Acetyl: 5-acet-1:3-xylidide. Needles from  
EtOH. M.p. 140.5° (144.5°).

Wroblewski, *Ann.*, 1881, **207**, 95.

Haller, Adams, Wherry, *J. Am. Chem.*  
*Soc.*, 1920, **42**, 184.

**p-Xylidine** (2-Amino-p-xylene)



$C_8H_{11}N$

MW, 121

Pale yellow leaflets. M.p. 15.5°. B.p. 213.5°.  
 $D_4^{21}$  0.9790.  $n_D^{21.5}$  1.55914. Mod. sol.  $H_2O$ .  
 $k = 9.63 \times 10^{-10}$  at 20°. Turns deep yellow in  
air.  $CrO_3 \rightarrow$  p-xyloquinone.

B,HCl: m.p. 228°.

Picrate: greenish-yellow. M.p. 171° decomp.

N-Me: see N-Methyl-p-xylidine.

N-Di-Me: see N-Dimethyl-p-xylidine.

N-Et:  $C_{10}H_{15}N$ . MW, 149. Oil. B.p. 222–  
3°/748 mm.

N-Benzyl:  $C_{16}H_{17}N$ . MW, 211. B.p. 320–5°.

N-Formyl: needles from  $H_2O$ . M.p. 116–17°.

N-Acetyl: 2-acet-1:4-xylidide. Needles from  
 $H_2O$  or toluene. M.p. 139°.

N-Chloroacetyl: needles from EtOH.Aq. M.p.  
153°.

N-Benzoyl: needles from EtOH. M.p. 140°.

N-p-Toluenesulphonyl: m.p. 232–3°.

Pflug, *Ann.*, 1889, **255**, 172.

Noelting, Witt, Forel, *Ber.*, 1885, **18**,  
2664.

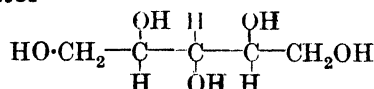
Jannasch, *Ann.*, 1875, **176**, 55.

Schmidt, E.P., 252,460, (*Chem. Abstracts*,  
1927, **21**, 2273).

**$\beta$ -Xylidinic Acid.**

See 4-Methylisophthalic Acid.

**Xylitol**



$C_5H_{12}O_5$

MW, 152

Syrup. Sweet taste.  $HI \rightarrow$  sec.-n-amyl  
iodide.  $PbO_2 + HCl \rightarrow$  xyloketose. Not  
oxidised by sorbose bacterium.

Dibenzylidene deriv.: cryst. M.p. 175°. Sol.  
 $CHCl_3$ . Prac. insol.  $H_2O$ , EtOH.

Penta-acetyl: m.p. 61.5–62.5°.

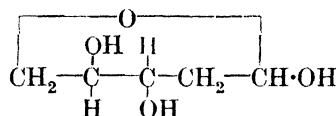
Fischer, *Ber.*, 1894, **27**, 2487.

Bertrand, *Bull. soc. chim.*, 1891, **5**, 555.

de Bruyn, v. Ekenstein, *Rec. trav. chim.*,  
1899, **18**, 151.

Bertrand, *Compt. rend.*, 1936, **203**, 143.

**2-Xylodesose**



$C_5H_{10}O_4$

MW, 134

d-.

Plates. M.p. 92–6°.  $[\alpha]_D^{22} - 40.25^\circ \rightarrow$   
 $+ 50.75^\circ$  in Py,  $- 22.5^\circ \rightarrow - 2.0^\circ$  in  $H_2O$ .  
Sol.  $H_2O$ , EtOH, Py. Spar. sol.  $Me_2CO$ . Insol.  
 $Et_2O$ ,  $CHCl_3$ ,  $CCl_4$ ,  $C_6H_6$ . Reduces Fehling's.

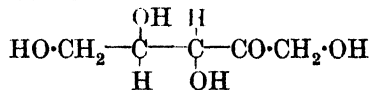
Benzylphenylhydrazones: prisms or plates.  
M.p. 116–18°.  $[\alpha]_D^{25} + 13.5^\circ$  in Py. Sol. EtOH,  
 $Me_2CO$ , Py. Spar. sol.  $Et_2O$ . Insol.  $H_2O$ .

Levene, Mori, *J. Biol. Chem.*, 1929, **83**,  
803.

**p-Xylohydroquinone.**

See 2:5-Dihydroxy-p-xylene.

**Xyloketose**



$C_5H_{10}O_5$

MW, 150

d-.

Syrup. Hygroscopic.  $[\alpha]_D^{25} - 33.2^\circ$ . Re-  
duces Fehling's. p-Bromophenylhydrazones: m.p.  
128–9°.  $[\alpha]_D^{20} + 23.7^\circ \rightarrow - 31.2^\circ$  in Py.

l-.

Free sugar not isolated. Reduces Fehling's.  
 $[\alpha]_D^{20} + 33.1^\circ$ .

Phenylosazone: m.p. 160–3°.  $[\alpha]_D + 0^\circ 15'$  in  
Py-EtOH. Identical with l-xylosazone and  
l-lyxosazone.

*p*-Bromophenylhydrazone: yellow plates from dil. EtOH. M.p. 130–1° (128°).  $[\alpha]_D^{20} - 25.8^\circ \rightarrow + 31.5^\circ$  in Py.  $[\alpha]_D - 1^\circ$  in EtOH.

dl.

Syrup. Sol. H<sub>2</sub>O.

Phenylosazone: m.p. 210–15°. Identical with dl-xylosazone.

Methylphenylosazone: yellow needles. M.p. 173°.

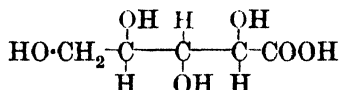
Neuberg, *Ber.*, 1902, **35**, 2628.

Levene, La Forge, *J. Biol. Chem.*, 1914, **18**, 319.

v. Vargha, *Ber.*, 1935, **68**, 24.

Schmidt, Treiber, *Ber.*, 1933, **66**, 1765.

### Xylonic Acid



C<sub>5</sub>H<sub>10</sub>O<sub>6</sub>

MW, 166

d.

Syrup.  $[\alpha]_D + 17.98^\circ$  (final) in H<sub>2</sub>O,  $- 1.9^\circ \rightarrow - 3.6^\circ \rightarrow + 20.2^\circ$  in H<sub>2</sub>O.

Cinchonine salt: plates from H<sub>2</sub>O or needles from EtOH. M.p. 180° decomp. (170°).  $[\alpha]_D^{17} + 125.0^\circ$  in H<sub>2</sub>O.

Brucine salt: cryst. from EtOH. M.p. 176° (172–4°).  $[\alpha]_D^{20} - 18.7^\circ$  in H<sub>2</sub>O.

Morphine salt: needles from dil. EtOH. M.p. 153°.

Amide: C<sub>5</sub>H<sub>11</sub>O<sub>5</sub>N. MW, 165. Plates. M.p. 81–2°.  $[\alpha]_D^{16} + 44.5^\circ \rightarrow + 23.8^\circ$  in H<sub>2</sub>O. Sol. H<sub>2</sub>O. Spar. sol. EtOH.

Phenylhydrazone: needles from AcOEt. M.p. 129° decomp.

$\gamma$ -Lactone: C<sub>5</sub>H<sub>8</sub>O<sub>5</sub>. MW, 148. Cryst. from Me<sub>2</sub>CO. M.p. 99–103° (98–101°, 90–2°).  $[\alpha]_D + 91.8^\circ \rightarrow + 86.7^\circ$  in H<sub>2</sub>O. Triacetyl: m.p. 99°.  $[\alpha]_D^{19} + 62.4^\circ$  in EtOH.

Tetra-acetyl: m.p. 86–8°.  $[\alpha]_D^{20} + 5^\circ$  in EtOH,  $- 2^\circ$  in CHCl<sub>3</sub>. Nitrile: laminæ from H<sub>2</sub>O. M.p. 81.5°.

2:3-Di-Me ether: obtained only in sol.  $[\alpha]_D^{22} + 30.4^\circ \rightarrow + 63^\circ$  in H<sub>2</sub>O. Phenylhydrazone: needles. M.p. 107–8°.  $[\alpha]_D^{23} + 30^\circ$  in EtOH. *p*-Bromophenylhydrazone: needles. M.p. 150–1°.  $\gamma$ -Lactone: syrup. B.p. about 115°/0.02 mm.  $[\alpha]_D^{22} + 97^\circ \rightarrow + 69^\circ$  in H<sub>2</sub>O.  $n_D^{19.5} 1.4640$ .

3:5-Di-Me ether: obtained only in sol. Phenylhydrazone: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 94–5°.  $\gamma$ -Lactone: syrup. B.p. 105–6°/0.08 mm.  $[\alpha]_D^{21.5} + 81.5^\circ \rightarrow + 85.1^\circ \rightarrow + 39^\circ$  in H<sub>2</sub>O.  $n_D^{19} 1.4643$ .

2:3:5-Tri-Me ether: obtained only in sol.  $[\alpha]_D^{17.80} + 42.5^\circ \rightarrow + 40^\circ \rightarrow + 62.5^\circ$  in H<sub>2</sub>O. Phenylhydrazone: needles from C<sub>6</sub>H<sub>6</sub>. M.p. 89–90°.  $\gamma$ -Lactone: syrup. B.p. 82°/0.06 mm.  $[\alpha]_D^{17.80} + 108^\circ \rightarrow + 110^\circ \rightarrow + 67.5^\circ$  in H<sub>2</sub>O.  $n_D^{17} 1.4464$ .

2:3:4-Tri-Me ether: obtained only in sol.  $[\alpha]_D^{16.461} + 32.7^\circ \rightarrow + 21.5^\circ$  in H<sub>2</sub>O. Phenylhydrazone: m.p. 137–138.5°.  $\delta$ -Lactone: needles from pet. ether. M.p. 56°.  $[\alpha]_D^{20} 0^\circ \rightarrow 21.5^\circ$  in H<sub>2</sub>O,  $[\alpha]_D^{15} - 3.8^\circ \rightarrow + 20.8^\circ$  in H<sub>2</sub>O.

l.

Free acid not isolated.

Tetra-acetyl: m.p. 86–8°.  $[\alpha]_D^{20} - 4.5^\circ$  in EtOH. Nitrile: cryst. from EtOH. M.p. 82°. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Prac. insol. H<sub>2</sub>O.

dl.

Free acid not isolated.

Tetra-acetyl: m.p. 134–5°.

Allen, Tollens, *Ann.*, 1890, **260**, 306.

Bertrand, *Bull. soc. chim.*, 1891, **5**, 556; 1896, **15**, 593.

Clowes, Tollens, *Ann.*, 1900, **310**, 175.

Maquenne, *Ann. chim. phys.*, 1901, **24**, 403.

Nef, Hedenburg, Glattfeld, *J. Am. Chem. Soc.*, 1917, **39**, 1650.

Haworth, Westgarth, *J. Chem. Soc.*, 1926, 880.

Haworth, Porter, *J. Chem. Soc.*, 1928, 616.

Hampton, Haworth, Hirst, *J. Chem. Soc.*, 1929, 1748.

Deulofeu, *J. Chem. Soc.*, 1929, 2459.

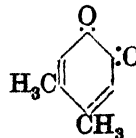
Isbell, Frush, *Chem. Abstracts*, 1934, **28**, 1667.

Major, Cook, *J. Am. Chem. Soc.*, 1936, **58**, 2476.

### Xylopicric Acid.

See 2:4:6-Trinitro-*m*-5-xylenol.

sym.-o-Xylo-o-quinone (4:5-Dimethyl-o-benzoquinone)



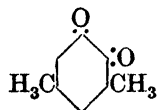
C<sub>8</sub>H<sub>8</sub>O<sub>2</sub>

MW, 136

Exist in two forms. (i) Red cryst. from Et<sub>2</sub>O. M.p. 102°. Spar. sol. C<sub>6</sub>H<sub>6</sub>, pet. ether. Sol. EtOH. (ii) Yellow plates.

Diepolder, *Ber.*, 1909, **42**, 2921.

**m-Xylo-o-quinone** (3 : 5-Dimethyl-o-benzoquinone)

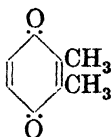


$C_8H_8O_2$  MW, 136

**Dioxime**: yellow needles from  $H_2O$ . M.p.  $142^\circ$  decomp. Sol. EtOH, AcOH,  $CHCl_3$ . Spar. sol.  $H_2O$ , petrol,  $C_6H_6$ .

Zincke, Schwarz, *Ann.*, 1899, 307, 48.

**o-Xylo-p-quinone** (2 : 3-Dimethyl-p-benzoquinone)



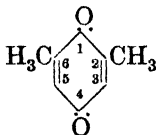
$C_8H_8O_2$  MW, 136

Yellow needles. M.p.  $55^\circ$ . Sublimes. Mod. sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

**Monoxime**: yellow needles. M.p.  $166^\circ$ . Very sol. MeOH, EtOH,  $C_6H_6$ ,  $NH_3$ . Spar. sol.  $H_2O$ .

Noelting, Forel, *Ber.*, 1885, 18, 2673.

**m-Xylo-p-quinone** (2 : 6-Dimethyl-p-benzoquinone)



$C_8H_8O_2$  MW, 136

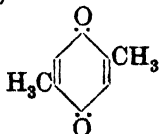
Yellow needles. M.p.  $72-3^\circ$ . Sublimes.

**1-Oxime**: yellow prisms from EtOH.Aq. M.p.  $175^\circ$ . Very sol. EtOH,  $Et_2O$ ,  $C_6H_6$ ,  $NH_3$ .

**4-Oxime**: yellow prisms from  $C_6H_6$ . M.p.  $170-1^\circ$ . Sol. EtOH, AcOH. Spar. sol. ligroin,  $C_6H_6$ . **Acetyl**: yellow cryst. from ligroin.

Noelting, Forel, *Ber.*, 1885, 18, 2679.

**p-Xylo-p-quinone** (2 : 5-Dimethyl-p-benzoquinone, phlorone)



$C_8H_8O_2$  MW, 136

Yellow needles from EtOH. M.p.  $125^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ . Mod. sol. EtOH. Spar. sol.  $H_2O$ . Sublimes.  $Zn + AcOH \rightarrow$  2 : 5-dihydroxy-p-xylene.

**Monoxime**: yellow needles from  $H_2O$ . M.p.

$168^\circ$ . Sol. EtOH,  $Et_2O$ ,  $C_6H_6$ . Very spar. sol.  $H_2O$ . **Me ether**: yellow cryst. M.p.  $70.5-71^\circ$ .

**Dioxime**: yellow needles from EtOH. M.p. about  $272^\circ$  ( $254^\circ$ ). Spar. sol. EtOH, AcOH,  $C_6H_6$ . Insol.  $H_2O$ . Sol. alkalis. **Diacyl**: yellow prisms from AcOH. M.p.  $170^\circ$ .

**Monobenzoylphenylhydrazine**: yellow prisms from ligroin. M.p.  $122-4^\circ$ ; solidifies to orange cryst., m.p.  $154-5^\circ$ .

Veibel, Simesen, *Ber.*, 1930, 63, 2480.

Auwers, Michaelis, *Ber.*, 1914, 47, 1289.

Carstanjen, *J. prakt. Chem.*, 1881, 23, 423.

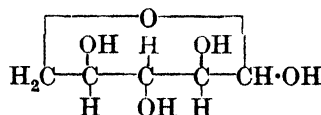
**m-Xylorcinol**.

See 4 : 6-Dihydroxy-m-xylene.

**p-Xylorcinol**.

See 2 : 6-Dihydroxy-p-xylene.

**Xylose** (Wood sugar)



$C_5H_{10}O_5$  MW, 150

*d.*

Needles. M.p.  $144-5^\circ$  ( $145-50^\circ$ ,  $153-4^\circ$ ,  $143^\circ$ ). Sweet taste.  $[\alpha]_D^{20} + 92.0^\circ \rightarrow +19.0^\circ$  in  $H_2O$ . Sol.  $H_2O$ , hot EtOH.  $D^{20} 1.535$ . Heat of comb.  $C_5$  3735 Cal. Reduces warm Fehling's. Does not ferment. Br water  $\rightarrow$  d-xylonic acid.  $HNO_3 \rightarrow$  xylotrihydroxyglutaric acid. Boil with dil.  $H_2SO_4 \rightarrow$  furfural.

**Semicarbazone**: m.p.  $202-4^\circ$ .  $[\alpha]_D^{20} - 38.8^\circ \rightarrow -24.4^\circ$  in  $H_2O$ . **Tetra-acetyl**: m.p.  $232-3^\circ$ .  $[\alpha]_D^{20} + 21^\circ$  in MeOH.

**Phenylosazone**: m.p.  $159^\circ$  ( $167^\circ$  decomp.,  $164^\circ$ ,  $161^\circ$ ).  $[\alpha]_D - 40.9^\circ$  in EtOH. Sol.  $Et_2O$ ,  $Me_2CO$ . Spar. sol.  $H_2O$ . Identical with d-lyxosazone.

**p-Bromophenylosazone**: yellow needles. M.p.  $208^\circ$  ( $204^\circ$ ). Insol.  $Me_2CO$ .

**o-Tolylosazone**: m.p.  $98-9^\circ$ .

**m-Tolylosazone**: m.p.  $137-8^\circ$ .

**p-Tolylosazone**: m.p.  $175-6^\circ$ .

**Phenylhydrazine**: yellow cryst.

**p-Bromophenylhydrazine**: yellow cryst. M.p.  $128^\circ$ .  $[\alpha]_D - 20.49^\circ$  in  $H_2O$ .

**2 : 4-Dibromophenylhydrazine**: m.p.  $127-8^\circ$ .

**p-Nitrophenylhydrazine**: deep yellow cryst. M.p.  $156^\circ$  ( $154-5^\circ$ ). Sol. EtOH.

**Methylphenylhydrazine**: laminæ from AcOEt. M.p.  $108-10^\circ$  ( $103-5^\circ$ ). Sol.  $H_2O$ . Insol.  $C_6H_6$ .

**Benzylphenylhydrazine**: needles. M.p.  $99^\circ$  ( $95-100^\circ$ ).  $[\alpha]_D - 33^\circ$  in EtOH. Sol. EtOH,  $Et_2O$ . Spar. sol.  $H_2O$ .

**Diphenylhydrazine**: yellow plates from dil.



EtOH or white needles from ligroin-Py. M.p. 128° (107-8°). Sol. hot H<sub>2</sub>O, EtOH, Me<sub>2</sub>CO, CHCl<sub>3</sub>. Insol. Et<sub>2</sub>O, ligroin.

**2-Naphthylhydrazone**: brown needles from MeOH. M.p. 123-4° (70°).  $[\alpha]_D^{20} + 18.6^\circ$  in MeOH,  $+ 15.8^\circ$  in AcOH. Sol. EtOH. Prac. insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>.

**$\alpha$ -Methylglucoside**:  $\alpha$ -methylxyloside. Cryst. M.p. 90-2°.  $[\alpha]_D^{20} + 153.2^\circ$  in H<sub>2</sub>O ( $+ 153.9^\circ$ ). **Triacetyl**: m.p. 86°.  $[\alpha]_D^{20} + 119.6^\circ$  in CHCl<sub>3</sub>. **2:3:4-Tri-Me ether**: b.p. 110°/10 mm.  $n_D^{25} 1.4397$ .  $[\alpha]_D^{20} + 121.5^\circ$  in CHCl<sub>3</sub>,  $+ 112.7^\circ$  in H<sub>2</sub>O,  $+ 122.2^\circ$  in MeOH.

**$\beta$ -Methylglucoside**:  $\beta$ -methylxyloside. Cryst. from AcOEt. M.p. 157° (155-6°).  $[\alpha]_D^{20} - 65.9^\circ$  in H<sub>2</sub>O. **Triacetyl**: m.p. 115°.  $[\alpha]_D^{20} - 60.8^\circ$  in CHCl<sub>3</sub>. **2:3:4-Tri-Me ether**: m.p. 51°. B.p. 69-72°/0.5 mm.  $[\alpha]_D^{20} - 69.5^\circ$  in CHCl<sub>3</sub>,  $- 81.7^\circ$  in H<sub>2</sub>O.

**$\gamma$ -Methylglucoside**:  $\gamma$ -methylxyloside. Syrup. B.p. 161.5°/0.03 mm.  $[\alpha]_D^{20} + 62.8^\circ$  in EtOH.

**2:3-Di-Me ether**: syrup.  $[\alpha]_D^{20} + 22.6^\circ \rightarrow + 24^\circ$  in H<sub>2</sub>O.  $n_D^{20} 1.4783$ .

**2:3:4-Tri-Me ether**: prisms from AcOEt. M.p. 91-2°.  $[\alpha]_D^{20} + 64.5^\circ \rightarrow + 17.7^\circ$  in H<sub>2</sub>O,  $+ 55.8^\circ \rightarrow + 24.2^\circ$  in CHCl<sub>3</sub>,  $+ 74^\circ \rightarrow + 21^\circ$  in EtOH.

**2:3:5-Tri-Me ether**: liq. B.p. 110°/0.04 mm.  $[\alpha]_D^{20} + 24.7^\circ \rightarrow + 31.2^\circ$  in H<sub>2</sub>O.

**Triacetyl**: cryst. from Et<sub>2</sub>O. M.p. 138-41°.  $[\alpha]_D^{25} + 70.11^\circ \rightarrow + 40.8^\circ$  in CHCl<sub>3</sub>.

**Tetra-acetyl**:  $\beta$ -form. M.p. 126-8°.  $[\alpha]_D^{21} - 24.4^\circ$  in CHCl<sub>3</sub>.

**Tetrabenzoyl**: m.p. 178°.  $[\alpha]_D^{22} - 47.45^\circ$  in Me<sub>2</sub>CO.

**Acetone deriv.**: m.p. 41-3°.  $[\alpha]_D^{18} - 19.0^\circ$ .

**Diacetone deriv.**: m.p. 44-5°. B.p. 85-7°/0.5 mm.  $[\alpha]_D^{22} + 13.0^\circ$  in H<sub>2</sub>O,  $+ 6.0^\circ$  in CHCl<sub>3</sub>.

*l.*

Needles or prisms. M.p. 144° (141-3°).  $[\alpha]_D^{20} - 79.3^\circ \rightarrow - 18.6^\circ$  in H<sub>2</sub>O.

**Tetra-acetyl**:  $\beta$ -form. M.p. 126°.  $[\alpha]_D^{25} + 25.7^\circ$  in CHCl<sub>3</sub>. **Aldehyde form**: m.p. 90-1°.  $[\alpha]_D^{20} + 22.5^\circ$  in CHCl<sub>3</sub>.

**Phenylosazone**: m.p. 159-61°.

*dl.*

Prisms. M.p. 129-31°.

**Phenylosazone**: yellow needles. M.p. 210-15° decomp. Identical with *dl*-lyxosazone.

Wheeler, Tollens, *Ann.*, 1889, **254**, 304.

Fischer, Stahel, *Ber.*, 1891, **24**, 528.

Schulze, Tollens, *Ann.*, 1892, **271**, 40.

Hudson, Harding, *J. Am. Chem. Soc.*, 1918, **40**, 1601.

Ling, Nanji, *J. Chem. Soc.*, 1923, **123**, 620.

Harding, *Sugar*, 1923, **25**, 124.

Carruthers, Hirst, *J. Chem. Soc.*, 1922, **121**, 2299.

Hirst, Purves, *J. Chem. Soc.*, 1923, **123**, 1352.

Haworth, Westgarth, *J. Chem. Soc.*, 1926, 880.

Hampton, Haworth, Hirst, *J. Chem. Soc.*, 1929, 1747.

Robertson, Speedie, *J. Chem. Soc.*, 1934, 824.

von Vargha, *Ber.*, 1935, **68**, 18.

Appel, *J. Chem. Soc.*, 1935, 425.

Sokolov, *Chem. Abstracts*, 1936, **30**, 5563.

### Xylosimine

C<sub>5</sub>H<sub>11</sub>O<sub>4</sub>N MW, 149

Cryst. M.p. 130° decomp.  $[\alpha]_D - 18^\circ 3' \rightarrow - 0^\circ 46'$  in H<sub>2</sub>O.

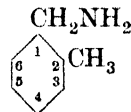
de Bruyn, van Leent, *Rec. trav. chim.*, 1895, **14**, 144.

Levene, *J. Biol. Chem.*, 1916, **24**, 61.

### Xylo-trihydroxyglutaric Acid.

See Trihydroxyglutaric Acid.

**o-Xylylamine** (*o*-Methylbenzylamine,  $\omega$ -amino-*o*-xylene)



C<sub>8</sub>H<sub>11</sub>N MW, 121

Oil. Cryst. in needles at  $- 20^\circ$ . B.p. 205.5-206°/745 mm., 125°/105 mm.  $D_4^{20} 0.9768$ .  $n_D^{19} 1.5436$ .

**B.HCl**: plates from H<sub>2</sub>O. M.p. 219-20°.

**B.HBr**: m.p. about 209°.

**Sulphate**: m.p. 176-9°.

**B.HNO<sub>3</sub>**: m.p. 130°.

**Oxalate**: m.p. 94-5°.

**B.HAuCl<sub>4</sub>**: m.p. about 180°.

**B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>**: yellow needles. M.p. 220-3° decomp.

**N-Acetyl**: needles from EtOH. M.p. 69°.

**N-Benzoyl**: needles from EtOH.Aq. M.p. 88°.

**Picrate**: yellow needles. M.p. 214-215.5°.

Konowalow, *J. Russ. Phys.-Chem. Soc.*, 1905, **37**, 536.

Strassmann, *Ber.*, 1888, **21**, 577.

**m-Xylylamine** (*m*-Methylbenzylamine,  $\omega$ -amino-*m*-xylene).

Liq. B.p. 205-205.5°/750.5 mm., 96°/20 mm. Sol. EtOH, Et<sub>2</sub>O. Insol. H<sub>2</sub>O.  $D_4^{20} 0.9654$ .

*B.HCl*: needles from EtOH. M.p. 208°.

*Sulphate*: m.p. 248° decomp.

*B<sub>2</sub>(COOH)<sub>2</sub>*: plates. M.p. 172°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: yellow plates. M.p. 214°.

*N-Acetyl*: liq. B.p. 235–40°.

*N-Benzoyl*: needles from CHCl<sub>3</sub>-ligroin. M.p. 150–150.5° (70°).

*Picrate*: cryst. from MeOH. M.p. 198° decomp. (156°).

Shoppee, *J. Chem. Soc.*, 1932, 701.

Rupe, Bernstein, *Helv. Chim. Acta*, 1930, 13, 462.

Sommer, *Ber.*, 1900, 33, 1074.

Brömme, *Ber.*, 1888, 21, 2701.

**p-Xylylamine** (p-Methylbenzylamine, ω-amino-p-xylene).

M.p. 12.6–13.2°. B.p. 204°/739 mm. *D*<sub>0</sub><sup>20</sup> 0.9520. *n*<sub>D</sub><sup>20</sup> 1.53639. Spar. sol. H<sub>2</sub>O.

*B.HCl*: needles. M.p. 234.5–235°.

*Sulphate*: plates from H<sub>2</sub>O. M.p. about 130°.

*B.HAuCl<sub>4</sub>*: yellow needles from H<sub>2</sub>O. M.p. 169–71°.

*B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>*: plates from H<sub>2</sub>O.

*B.HSnCl<sub>3</sub>*: m.p. 107°.

*N-Acetyl*: cryst. from EtOH. M.p. 107–8°.

*N-Benzoyl*: needles from EtOH. M.p. 137°.

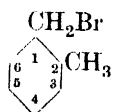
*Picrate*: cryst. from H<sub>2</sub>O. M.p. 204° decomp.

Druce, *J. Chem. Soc.*, 1918, 113, 718.

Curtius, Darapsky, *Ber.*, 1902, 35, 3232.

Lustig, *Ber.*, 1895, 28, 2988.

**o-Xylyl bromide** (o-Methylbenzyl bromide, ω-bromo-o-xylene)



C<sub>8</sub>H<sub>9</sub>Br

MW, 185

Prisms. M.p. 21°. B.p. 216–17°/742 mm., 108°/16 mm. *D*<sub>25</sub><sup>25</sup> 1.3811.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, 91, 1695.

**m-Xylyl bromide** (m-Methylbenzyl bromide, ω-bromo-m-xylene).

Liq. B.p. 212.5° (slight decomp.), 185°/340 mm., 100–1°/14 mm. *D*<sub>25</sub><sup>25</sup> 1.3711.

Titley, *J. Chem. Soc.*, 1926, 514.

Haller, Bauer, *Compt. rend.*, 1911, 153, 22.

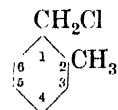
Atkinson, Thorpe, *J. Chem. Soc.*, 1907, 91, 1696.

**p-Xylyl bromide** (p-Methylbenzyl bromide, ω-bromo-p-xylene).

Needles from EtOH. M.p. 35°. B.p. 218–20°/740 mm. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, 91, 1697.

**o-Xylyl chloride** (o-Methylbenzyl chloride, ω-chloro-o-xylene)



(C<sub>8</sub>H<sub>9</sub>Cl)

MW, 140.5

Liq. B.p. 197–9° (195–203°).

Reyman, *Bull. soc. chim.*, 1876, 26, 534.

**m-Xylyl chloride** (m-Methylbenzyl chloride, ω-chloro-m-xylene).

Liq. B.p. 195–6°. *D*<sub>20</sub><sup>20</sup> 1.064. *n*<sub>D</sub><sup>25</sup> 1.5327.

King, Merriam, *Chem. Zentr.*, 1935, II, 2359.

Gundelach, *Bull. soc. chim.*, 1876, 26, 43.

**p-Xylyl chloride** (p-Methylbenzyl chloride, ω-chloro-p-xylene).

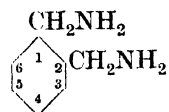
Fuming liq. B.p. 200–2°, 90°/20 mm.

Curtius, Sprenger, *J. prakt. Chem.*, 1900, 62, 111.

**Xylyl cyanide.**

See under Tolylacetic Acid.

**o-Xylylenediamine** (ω-Diamino-o-xylene)



C<sub>8</sub>H<sub>12</sub>N<sub>2</sub>

MW, 136

Liq. with ammoniacal odour. Absorbs CO<sub>2</sub> from the air.

*N-Tetra-Me*: C<sub>12</sub>H<sub>20</sub>N<sub>2</sub>. MW, 192. Liq. B.p. 105–6°/14 mm. *Dimethiodide*: m.p. 219° decomp. *Dimethobromide*: prisms from EtOH-Et<sub>2</sub>O. M.p. 207–8°. *Picrate*: yellow cryst. M.p. 187–8°.

*N-Tetra-Et*: C<sub>16</sub>H<sub>28</sub>N<sub>2</sub>. MW, 248. Liq. B.p. 170–5°/20 mm.

*N:N'-Diphenyl*: C<sub>20</sub>H<sub>20</sub>N<sub>2</sub>. MW, 288. Plates from EtOH. M.p. 114°. Sol. EtOH, C<sub>6</sub>H<sub>6</sub>. Insol. H<sub>2</sub>O, ligroin. *N:N'-Di-Me*: C<sub>22</sub>H<sub>24</sub>N<sub>2</sub>. MW, 316. Plates from EtOH. M.p. 110°.

*N-Tetraphenyl*: C<sub>32</sub>H<sub>28</sub>N<sub>2</sub>. MW, 440. Needles from Me<sub>2</sub>CO-Aq. M.p. 179°. Sol. Me<sub>2</sub>CO, CHCl<sub>3</sub>. Spar. sol. EtOH.

N: N'-*Diacetyl*: cryst. from Et<sub>2</sub>O. M.p. 146°.

N: N'-*Dibenzoyl*: needles from EtOH. M.p. 184°.

*Picrate*: yellow needles. Decomp. at 170°.

Gabriel, Pinkus, *Ber.*, 1893, 26, 2212.

Strassmann, *Ber.*, 1888, 21, 579.

**m-Xylylenediamine** (*ω*-*Diamino-m-xylene*).

Liq. B.p. 245-8°. Misc. with EtOH, Et<sub>2</sub>O.

N: N'-*Diacetyl*: cryst. M.p. 118-19°.

*Picrate*: yellow plates. Decomp. at 185-90°.

Brömme, *Ber.*, 1888, 21, 2705.

**p-Xylylenediamine** (*ω*-*Diamino-p-xylene*).

Cryst. M.p. 35°.

B<sub>2</sub>H<sub>2</sub>PtCl<sub>6</sub>: cryst. Decomp. at 250°.

N-*Tetraphenyl*: C<sub>32</sub>H<sub>28</sub>N<sub>2</sub>. MW, 440. Needles from AcOH. M.p. 186°. Sol. AcOH. Mod. sol. Me<sub>2</sub>CO. Spar. sol. EtOH.

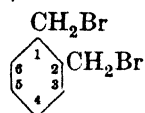
N-*Tetra-acetyl*: needles from EtOH. M.p. 194°.

N: N'-*Dibenzoyl*: needles. M.p. 193-4°.

*Picrate*: orange needles. Decomp. at 232°.

Lustig, *Ber.*, 1895, 28, 2992.

**o-Xylylene dibromide** (*o*-*Xylylene bromide*, *ω*-*dibromo-o-xylene*)



C<sub>8</sub>H<sub>8</sub>Br<sub>2</sub>

MW, 264

Cryst. from CHCl<sub>3</sub>. M.p. 95°. Decomp. on dist. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>. Mod. sol. pet. ether.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, 91, 1696.

**m-Xylylene dibromide** (*m*-*Xylylene bromide*, *ω*-*dibromo-m-xylene*).

Prisms from Me<sub>2</sub>CO. M.p. 77°. B.p. 158-60°/12 mm. Sol. Et<sub>2</sub>O, CHCl<sub>3</sub>, ligroin. D<sub>20</sub> 1.959.

Titely, *J. Chem. Soc.*, 1926, 514.

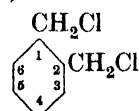
Braun, Karpf, Garn, *Ber.*, 1920, 53, 101.

**p-Xylylene dibromide** (*p*-*Xylylene bromide*, *ω*-*dibromo-p-xylene*).

Cryst. from CHCl<sub>3</sub> or C<sub>6</sub>H<sub>6</sub>. M.p. 145-7°. B.p. 245°. D<sub>20</sub> 2.012. Sol. CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O.

Atkinson, Thorpe, *J. Chem. Soc.*, 1907, 91, 1698.

**o-Xylylene dichloride** (*o*-*Xylylene chloride*, *ω*-*dichloro-o-xylene*)



C<sub>8</sub>H<sub>8</sub>Cl<sub>2</sub>

MW, 175

Cryst. from pet. ether. M.p. 55°. B.p. 239-41°, 130-5°/19 mm. Sol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>, ligroin. D<sub>20</sub> 1.393.

Quelet, *Bull. soc. chim.*, 1933, 53, 222.

**m-Xylylene dichloride** (*m*-*Xylylene chloride*, *ω*-*dichloro-m-xylene*).

Cryst. M.p. 34-2°. B.p. 250-5°. D<sub>20</sub> 1.302.

Colson, Gautier, *Ann. chim.*, 1887, 11, 23.

**p-Xylylene dichloride** (*p*-*Xylylene chloride*, *ω*-*dichloro-p-xylene*).

Plates from EtOH. M.p. 100°. B.p. 240-5° decomp., 135°/16 mm. D<sub>20</sub> 1.417.

Quelet, *Bull. soc. chim.*, 1933, 53, 222.

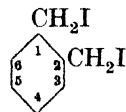
Sabetay, *Compt. rend.*, 1931, 192, 1109.

Grimaux, *Ann.*, 1870, 155, 340.

**Xylylene dicyanide.**

See under Phenylenediacetic Acid.

**o-Xylylene di-iodide** (*o*-*Xylylene iodide*, *ω*-*di-iodo-o-xylene*)



C<sub>8</sub>H<sub>8</sub>I<sub>2</sub>

MW, 358

Yellow prisms from Et<sub>2</sub>O. M.p. 109-10°.

Finkelstein, *Ber.*, 1910, 43, 1532.

**m-Xylylene di-iodide** (*m*-*Xylylene iodide*, *ω*-*di-iodo-m-xylene*).

M.p. 106°.

See previous reference.

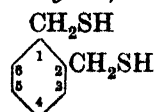
**p-Xylylene di-iodide** (*p*-*Xylylene iodide*, *ω*-*di-iodo-p-xylene*).

M.p. 175° decomp. Sol. hot EtOH, CHCl<sub>3</sub>. Spar. sol. Et<sub>2</sub>O.

Knoll, D.R.P., 230,172, (*Chem. Zentr.*, 1911, I, 359).

Finkelstein, *Ber.*, 1910, 43, 1532.

**o-Xylylene Dimercaptan** (*Dithio-o-xylylene glycol*, *ω*-*dimercapto-o-xylene*)



C<sub>8</sub>H<sub>10</sub>S<sub>2</sub>

MW, 170

Prisms. M.p. 45–6°. B.p. 160°/20 mm.  
Sol. EtOH, Et<sub>2</sub>O. Spar. sol. pet. ether.

Autenrieth, Hennings, *Ber.*, 1901, **34**, 1774.

**m-Xylylene Dimercaptan** (*Dithio-m-xylylene glycol, ω-dimercapto-m-xylene*).

Liq. with characteristic odour. B.p. 157–8°/15 mm.

*Dibenzoyl*: prisms. M.p. 52.5°.

Autenrieth, Beuttel, *Ber.*, 1909, **42**, 4358.

**p-Xylylene Dimercaptan** (*Dithio-p-xylylene glycol, ω-dimercapto-p-xylene*).

Cryst. M.p. 46–7°. B.p. 156°/12 mm.

*Dibenzoyl*: needles. M.p. 135°.

Autenrieth, Beuttel, *Ber.*, 1909, **42**, 4349.

**o-Xylylene oxide.**

See Phthalan.

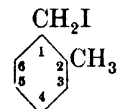
**Xylylic Acid.**

See Dimethylbenzoic Acid.

**Xylylidene chloride.**

See Methylbenzylidene chloride.

**o-Xylyl iodide** (*o-Methylbenzyl iodide, ω-iodo-o-xylene*)



C<sub>8</sub>H<sub>9</sub>I

MW, 232

Needles from Et<sub>2</sub>O. M.p. 34°. Decomp. on dist.

Zeltner, Tarassow, *Ber.*, 1910, **43**, 945.

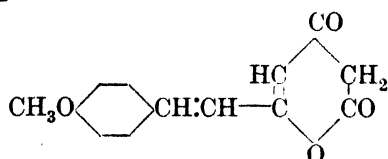
**p-Xylyl iodide** (*p-Methylbenzyl iodide, ω-iodo-p-xylene*).

Needles from Et<sub>2</sub>O. M.p. 45.5–46.5°.

Zeltner, Tarassow, *Ber.*, 1910, **43**, 944.

## Y

**Yangonalactone**



C<sub>14</sub>H<sub>12</sub>O<sub>4</sub>

MW, 244

Yellow prisms from MeOH or EtOH. M.p. 238°. Sol. Et<sub>2</sub>O, AcOEt, AcOH, C<sub>6</sub>H<sub>6</sub>. Forms derivs. of enolic form.

*Me ether*: see Yangonin.

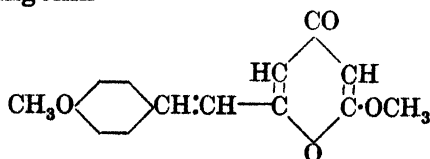
*O-Acetyl*: red prisms from AcOEt. M.p. 133°.

*O-Benzoyl*: yellow leaflets from EtOH. M.p. 147°.

Borsche, Blount, *Ber.*, 1932, **65**, 820.

Borsche, Bodenstein, *Ber.*, 1929, **62**, 2519.

**Yangonin**



C<sub>15</sub>H<sub>14</sub>O<sub>4</sub>

MW, 258

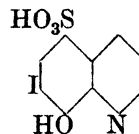
Constituent of Kawa root. Greenish-yellow cryst. with blue fluor. from MeOH or Me<sub>2</sub>CO. M.p. 153–4°. Sol. Me<sub>2</sub>CO, AcOH. Spar. sol. Et<sub>2</sub>O, CS<sub>2</sub>. Sol. conc. H<sub>2</sub>SO<sub>4</sub> → yellow sol. with green fluor.

Jablonski, *Chem. Abstracts*, 1935, **29**, 7982.

Borsche, Bodenstein, *Ber.*, 1929, **62**, 2519.

Murakami, *Chem. Abstracts*, 1918, **12**, 2547.

**Yatren** (*Loretin, quinoxyl, 7-iodo-8-hydroxy-quinoline-5-sulphonic acid*)



C<sub>9</sub>H<sub>6</sub>O<sub>4</sub>NIS

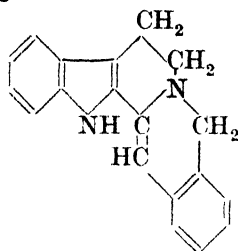
MW, 351

Yellow prisms or laminæ. Decomp. at about 260°. Very spar. sol. H<sub>2</sub>O, EtOH. Prac. insol. Et<sub>2</sub>O, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>. Sol. conc. H<sub>2</sub>SO<sub>4</sub>. HNO<sub>3</sub> (D 1.52) → 5:7-dinitro-8-hydroxyquinoline. Forms cryst. salts with Na, K, Mg, Ca, Ba, Sr. Used as disinfectant in treatment of amœbic dysentery, etc.

I.G., D.R.P., 545,915, (*Chem. Abstracts*, 1933, **27**, 566).

Claus, *Arch. Pharm.*, 1893, **231**, 706.

## Yobyrine

 $C_{19}H_{16}N_2$ 

MW, 272

Cryst. from  $C_6H_6$  or EtOH. M.p. 218–19°.Wibaut, Gastel, *Rec. trav. chim.*, 1935, **54**, 85.Barger, Scholz, *Helv. Chim. Acta*, 1933, **16**, 1346.

## Yohimbene

 $C_{21}H_{26}O_3N_2$ 

MW, 354

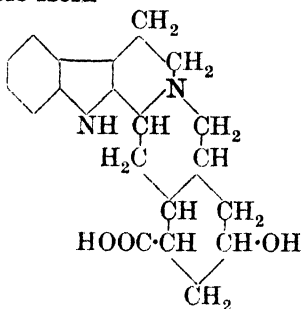
Isomeric with yohimbine. Leaflets from MeOH. M.p. 278° decomp. Sol. MeOH, EtOH. Spar. sol.  $Me_2CO$ ,  $CHCl_3$ ,  $Et_2O$ . Insol. AcOEt, petrol,  $C_6H_6$ , toluene.  $[\alpha] + 43.7^\circ$  in Py. Hyd. with loss of  $CO_2 \rightarrow$  yohimbol. Extremely poisonous.

*B,HCl*: needles +  $3H_2O$  from MeOH.Aq. M.p. 234°.  $[\alpha] - 8.8^\circ$  in  $H_2O$ .

*B,MeI*: needles +  $4H_2O$  from  $Et_2O$ -EtOH. Decomp. at 288°.

Hahn, Stenner, *Ber.*, 1928, **61**, 282.Hahn, Brandenburg, *Ber.*, 1926, **59**, 2189.

## Yohimbic Acid



Suggested formula

 $C_{20}H_{24}O_3N_2$ 

MW, 340

Cryst. +  $1H_2O$  from  $H_2O$ . M.p. 256° (265–9°).  $[\alpha]_D^{20} + 138.8^\circ$  in Py. Loss of  $CO_2 \rightarrow$  yohimbol.

*Me ester*: see Yohimbine.

*Et ester*:  $C_{22}H_{28}O_3N_2$ . MW, 368. Cryst. M.p. 190°.

*Propyl ester*:  $C_{23}H_{30}O_3N_2$ . MW, 382. Cryst. +  $1H_2O$ . M.p. 137°.

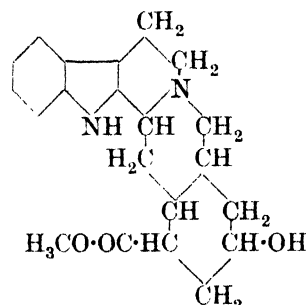
*Butyl ester*:  $C_{24}H_{32}O_3N_2$ . MW, 396. Cryst. +  $1H_2O$ . M.p. 119–22°, anhyd. 127°.

*Benzyl ester*:  $C_{27}H_{30}O_3N_2$ . MW, 430. Amorp. powder from EtOH.Aq. M.p. 77–8°. *B,HCl*: cryst. powder from EtOH- $Me_2CO$ . M.p. 253–4°.

Worrall, *J. Am. Chem. Soc.*, 1935, **57**, 900; 1933, **55**, 3715.

Field, *J. Chem. Soc.*, 1923, **123**, 3003.

**Yohimbine** (*Quebrachine*, *hydroergotocin*, *aphrodine*, *corynine*, methyl ester of yohimbic acid)



Suggested formula

 $C_{21}H_{26}O_3N_2$ 

MW, 354

Chief alkaloid of *Corynanthe Johimbe*. Needles from EtOH.Aq. M.p. 235°. Sol. EtOH,  $CHCl_3$ , hot  $C_6H_6$ . Mod. sol.  $Et_2O$ . Spar. sol.  $H_2O$ .  $[\alpha]_D^{20} + 107.9^\circ$  in Py.

*B,HCl*: plates. M.p. 302°.  $[\alpha]_D^{23} + 103.3^\circ$  in  $H_2O$ .

*Nitrate*: m.p. 276°.

*Mono-acetyl*: m.p. 150°. Sol. EtOH,  $Et_2O$ ,  $CHCl_3$  with green fluor.

*O:N-Diacetyl*: m.p. 183°.

*Methiodide*: plates from  $Me_2CO$ . M.p. 249–50°.

Robinson, *Annual Reviews on Biochemistry*, 1935, **4**, 504.

Warnat, *Ber.*, 1930, **63**, 2959.

Schomer, *Chem. Zentr.*, 1927, **II**, 2309.

Field, *J. Chem. Soc.*, 1923, **123**, 3003.

Isomeric forms of yohimbine are described in Heinemann, *Ber.*, 1934, **67**, 18.

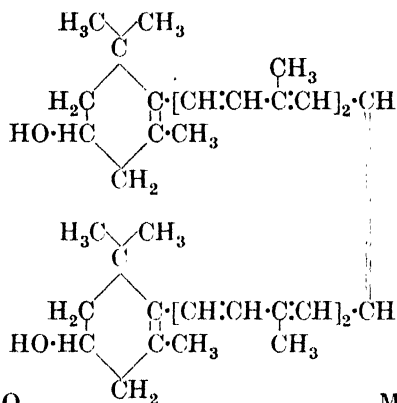
**Yperite.**

See 2:2'-Dichlorodiethyl sulphide.

## Z

**Zanthotoxin.**

See Xanthotoxin.

**Zeaxanthin** $C_{40}H_{56}O_2$ 

MW, 568

Constituent of lipochrome of *Fucus vesiculosus*, *Physalis*, egg yolk and spindle-tree maize. Yellow prisms from MeOH, cryst. +  $\frac{1}{2}$  MeOH from MeOH- $C_6H_6$ . M.p.  $206.5^\circ$  ( $215.5^\circ$  corr.). Mod. sol.  $Et_2O$ ,  $CS_2$ ,  $C_6H_6$ ,  $CHCl_3$ ,  $CCl_4$ , Py, AcOEt. Spar. sol. pet. ether, MeOH. Absorption maxima at 515 m $\mu$  and 485 m $\mu$  in  $CS_2$ .

*Mono-Me ether*:  $C_{41}H_{58}O_2$ . MW, 582. Needles from MeOH. M.p.  $153^\circ$ .

*Di-Me ether*:  $C_{42}H_{60}O_2$ . MW, 596. Cryst. from MeOH. M.p.  $176^\circ$ .

*Diacetyl*: m.p.  $154^\circ$ .

*Dipropionyl*: cryst. from  $C_6H_6$ -MeOH. M.p.  $142^\circ$ .

*Dibutyl*: cryst. from  $C_6H_6$ -MeOH. M.p.  $132^\circ$ .

*Divaleryl*: cryst. from  $C_6H_6$ -MeOH. M.p.  $125^\circ$ .

*Dicaproyl*: cryst. from  $C_6H_6$ -MeOH. M.p.  $117-18^\circ$ .

*Dicaprylyl*: cryst. from  $C_6H_6$ . M.p.  $107^\circ$ .

*Dilauryl*: m.p.  $104^\circ$ .

*Monopalmityl*: plates from  $C_6H_6$ -EtOH. M.p.  $148^\circ$ .

*Dipalmityl*: cryst. from  $C_6H_6$ -MeOH. M.p.  $99.5^\circ$ .

*Distearyl*: m.p.  $95^\circ$ .

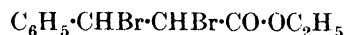
Karrer, Solmssen, *Helv. Chim. Acta*, 1935, **18**, 477.

Heilbron, Phipers, *Biochem. J.*, 1935, **29**, 1369.

Gillam, *ibid.*, 1831.

Kuhn, Grundmann, *Ber.*, 1934, **67**, 596.

Karrer, Wehrli, Helfenstein, *Helv. Chim. Acta*, 1930, **13**, 271.

**Zebromal** (Ethyl ester of trans- $\alpha$ : $\beta$ -dibromo-cinnamic acid) $C_{11}H_{12}O_2Br_2$ 

MW, 336

Plates. M.p.  $75-6^\circ$ . Sol.  $Et_2O$ ,  $CHCl_3$ . Spar. sol. EtOH. Insol.  $H_2O$ . Boiling alc. KOH  $\rightarrow$  phenylpropionic acid. Used in treatment of epilepsy.

Merck, D.R.P., 271,434, (*Chem. Zentr.*, 1914, **1**, 1235).

Merck, *Pharmazeutische Zentralhalle*, 1912, **53**, 591.

Anschütz, Kinnicutt, *Ber.*, 1878, **11**, 1220.

**Zierin** (Glucoside of cyanhydrin of m-hydroxybenzaldehyde) $C_{14}H_{17}O_7N$ 

MW, 311

Obtained from aerial parts of *Zieria laevigata*. Needles from AcOEt- $CHCl_3$ . Softens at  $153^\circ$ , m.p.  $156^\circ$ . Sol.  $H_2O$ , EtOH,  $Me_2CO$ , MeCOEt. Very spar. sol.  $Et_2O$ ,  $CHCl_3$ , pet. ether,  $C_6H_6$ .  $[\alpha]_D^{20} = 29.5^\circ$ .  $FeCl_3 \rightarrow$  faint blue col. Hyd.  $\rightarrow$  glucose + HCN + m-hydroxybenzaldehyde.

*Acetyl*: cryst. from EtOH.Aq. M.p.  $115-18^\circ$ .

Finnemore, Cooper, *J. Proc. Roy. Soc. New South Wales*, 1936, **70**, 175.

(*Chem. Zentr.*, 1937, **1**, 878).

**Zierone** $C_{15}H_{22}O$ 

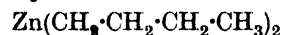
MW, 218

Constituent of volatile oil of *Zieria macrophylla*, Bonpland. Sesquiterpene ketone, isomeric with eremophilone. Viscous colourless oil. B.p.  $147-9^\circ/18$  mm.

*Semicarbazone*: prisms from MeOH. M.p.  $182^\circ$ .

*2:4-Dinitrophenylhydrazones*: needles from EtOH. M.p.  $95-7^\circ$ .

Bradfield, Penfold, Simonsen, *J. Proc. Roy. Soc. New South Wales*, 1933, **67**, 200, (*Chem. Zentr.*, 1934, **1**, 1982).

**Zinc dibutyl** $C_8H_{18}Zn$ 

MW, 179.5

Liq. B.p.  $81-2^\circ/9$  mm.

Noller, *Organic Syntheses*, 1932, **XII**, 86.

**Zinc diethyl (Zinc ethyl)**
 $\text{C}_4\text{H}_{10}\text{Zn}$ 

MW, 123.5

M.p.  $-28^\circ$ . B.p.  $118^\circ$ .  $D^{18}_4$  1.1826. Spontaneously inflammable in air.  $\text{H}_2\text{O} \rightarrow \text{C}_2\text{H}_6 + \text{Zn}(\text{OH})_2$ .  $\text{NH}_3 \rightarrow \text{Zn}(\text{NH}_2)_2$ . Hydroxy compounds  $\rightarrow$  ethane.

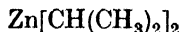
See previous reference.

**Zinc di-isobutyl**
 $\text{C}_8\text{H}_{18}\text{Zn}$ 

MW, 179.5

Liq. B.p.  $185^\circ$  ( $165-7^\circ$ ).

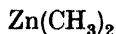
Ponzio, *Gazz. chim. ital.*, 1900, **30**, ii, 25.

**Zinc di-isopropyl**
 $\text{C}_6\text{H}_{14}\text{Zn}$ 

MW, 151.5

Liq. B.p.  $135^\circ$ ,  $94-8^\circ/40$  mm. Fumes in air. Inflammable with difficulty in air and oxidised to  $\text{Zn}(\text{OC}_3\text{H}_7)_2$ .

Bohm, *Chem. Zentr.*, 1899, I, 1067.

**Zinc dimethyl (Zinc methyl)**
 $\text{C}_2\text{H}_6\text{Zn}$ 

MW, 95.5

M.p.  $-42.5^\circ$ . B.p.  $46^\circ$ .  $D^{10}_4$  1.386. Ignites spontaneously in air.  $\text{H}_2\text{O} \rightarrow \text{CH}_4 + \text{Zn}(\text{OH})_2$ .

Renshaw, Greenlaw, *J. Am. Chem. Soc.*, 1920, **42**, 1472.

**Zinc diphenyl**
 $\text{C}_{12}\text{H}_{10}\text{Zn}$ 

MW, 219.5

Needles from  $\text{C}_6\text{H}_6$ . M.p.  $105-6^\circ$ . B.p.  $280-5^\circ$  slight decomp. Sol.  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ . Spar. sol. pet. ether. Decomp. in dry air  $\rightarrow \text{ZnO} +$  diphenyl.  $\text{H}_2\text{O} \rightarrow \text{Zn}(\text{OH})_2 + \text{C}_6\text{H}_6$ .  $\text{CHCl}_3 \rightarrow$  triphenylmethane.

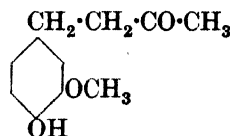
Hilpert, Grüttner, *Ber.*, 1913, **46**, 1680.

**Zinc dipropyl (Zinc propyl)**
 $\text{C}_6\text{H}_{14}\text{Zn}$ 

MW, 151.5

Liq. B.p.  $146^\circ$ ,  $39-40^\circ/9$  mm.  $\text{H}_2\text{O} \rightarrow$  propane +  $\text{Zn}(\text{OH})_2$ .

Noller, *Organic Syntheses*, 1932, XII, 86.

**Zingerone (Zingiberone, 4-hydroxy-3-methoxybenzylacetone)**
 $\text{C}_{11}\text{H}_{14}\text{O}_3$ 

MW, 294

Constituent of oil of ginger. Needles or plates with sweet odour from  $\text{Et}_2\text{O}$ -pet. ether. M.p.  $40-1^\circ$ . Spar. sol.  $\text{H}_2\text{O}$ , pet. ether. Sol. dil. alkalis. Slowly volatile in steam. Reduces  $\text{NH}_3\cdot\text{AgNO}_3$  in the warm. Alc.  $\text{FeCl}_3 \rightarrow$  green col.

*Oxime*: m.p.  $87.5-88.5^\circ$ .

*Semicarbazone*: needles. M.p. about  $133^\circ$ .

*Phenylhydrazone*: plates. M.p. about  $143^\circ$ .

*Me ether*:  $\text{C}_{12}\text{H}_{16}\text{O}_3$ . MW, 308. Needles from  $\text{EtOH}$  or  $\text{MeOH}$ . M.p.  $55-6^\circ$ . B.p.  $186^\circ/16$  mm. Spar. sol. pet. ether. Insol.  $\text{H}_2\text{O}$ . *Oxime*: needles from  $\text{Et}_2\text{O}$ -pet. ether or  $\text{MeOH}$ . Aq. M.p.  $93-4^\circ$ .

*Et ether*:  $\text{C}_{13}\text{H}_{18}\text{O}_3$ . MW, 322. Cryst. from  $\text{EtOH}$ . Aq. or pet. ether. M.p.  $66^\circ$ . Insol.  $\text{H}_2\text{O}$ .

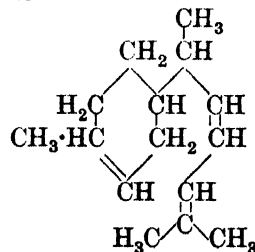
*Acetyl*: cryst. M.p.  $40-2^\circ$ . B.p.  $204-5^\circ/14$  mm.

*Benzoyl*: cryst. from  $\text{EtOH}$ . M.p.  $126-7^\circ$ .

Mannich, Merz, *Chem. Abstracts*, 1927, **21**, 1449.

Nomura, Canadian P., 203,512 (*Chem. Abstracts*, 1920, **14**, 2936).

Lapworth, Pearson, Royle, *J. Chem. Soc.*, 1917, **111**, 777.

**Zingiberene**
 $\text{C}_{15}\text{H}_{24}$ 

MW, 204

Constituent of ginger oil. Natural oil is always contaminated with bisabolene. B.p.  $134^\circ/14$  mm.  $D^{20}_4$  0.8684.  $n^{20}_D$  1.4956.  $[\alpha]^{20}_D -73.38^\circ$ . Resinifies on standing.  $\text{HBr} \rightarrow$  dihydrobromide of isozingiberene.  $\text{AcOH} + \text{H}_2\text{SO}_4 \rightarrow$  isozingiberene.  $\text{AcOH} (+ \text{Pt}) \rightarrow$  hexahydrozingiberene. Heat with S  $\rightarrow$  cadalene.

*Nitrosochloride*: m.p.  $93-4^\circ$ .

*Nitrosite* : (i) M.p. 120–1°. (ii) M.p. 105°.

*Nitrosate* : m.p. 86–8°.

Ruzicka, van Veen, *Ann.*, 1929, **468**, 143.

Moudgill, *J. Indian Chem. Soc.*, 1928, **5**, 255.

### Zingiberol

$C_{15}H_{26}O$

MW, 222

Constituent of ginger oil. Liq. B.p. 154–7°/14.5 mm. HCl in AcOH  $\rightarrow$  isozingiberene dihydrochloride.

Brooks, *J. Am. Chem. Soc.*, 1916, **38**, 431.

### Zingiberone.

*See* Zingerone.

### Zoomaric Acid.

*See* Palmitoleic Acid.

### Zygadenine

$C_{39}H_{63}O_{10}N$

MW, 705

Alkaloid from leaves of *Zygadenus intermedius* (Death camas). Cryst. from  $Et_2O$ , needles from  $C_6H_6$ , prisms + 2EtOH from EtOH. M.p. 200–1°.  $[\alpha] - 48.2^\circ$  in  $CHCl_3$ . Conc.  $H_2SO_4 \rightarrow$  orange to red col. Physiological action resembles that of cevadine.

Heyl, Hepner, Loy, *J. Am. Chem. Soc.*, 1913, **35**, 258.





# DICTIONARY OF ORGANIC COMPOUNDS

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# 1943 SUPPLEMENT TO VOLUME III

## N

### Napelline

$C_{22}H_{33}O_3N$  MW, 359

Alkaloid of *Aconitum napellus*. Plates from  $Et_2O$ -pet. ether or  $Me_2CO$ .Aq.

$B, HCl$ : m.p.  $220-2^\circ$  decomp.  $[\alpha]_D^{22} - 93.9^\circ$  in  $H_2O$ .

$B, HBr$ : needles from  $MeOH-Et_2O$ . M.p.  $229^\circ$  decomp.  $[\alpha]_D^{22} - 42.7^\circ$  in  $H_2O$ .

$B, HI$ : cryst. from  $MeOH-Et_2O$ . M.p.  $181.5^\circ$  decomp.

Freudenberg, Rogers, *J. Am. Chem. Soc.*, 1937, 59, 2572.

### Naphthacene.

See 2:3-Benzanthracene.

### Naphthacenequinone.

See 2:3-Benzanthraquinone.

### Naphthaflavone.

See Benzflavone.

### Naphthaflavonol.

See Benzflavonol.

### Naphthalepidine.

See Methylbenzquinoline.

### Naphthanthracene.

See 1:2-Benzanthracene.

### Naphthanthraquinone.

See 1:2-Benzanthraquinone.

### Naphthaphenazine.

See Benzphenazine.

### Naphthapyridine.

See Benzquinoline and Anthrapyridine.

### Naphthaquinaldine.

See Methylbenzquinoline.

### Naphthaquinoline.

See Benzquinoline.

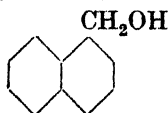
### Naphthazine.

See  $\alpha$ -Anthrapyridine.

### Naphthylbutane.

See Butylnaphthalene.

**1-Naphthylcarbinol** ( $\alpha$ -Menaphthyl alcohol, 1-hydroxymethylnaphthalene)



$C_{11}H_{10}O$

MW, 158

Needles from  $H_2O$ . M.p.  $59.5-60^\circ$ . B.p.  $301^\circ/715\text{ mm.}$ ,  $163-6^\circ/15\text{ mm.}$

Acetyl: b.p.  $172-3^\circ/13\text{ mm.}$

Ziegler, *Ber.*, 1921, 54, 739.

**2-Naphthylcarbinol** ( $\beta$ -Menaphthyl alcohol, 2-hydroxymethylnaphthalene).

Leaflets. M.p.  $90.5^\circ$  1. cold  $H_2O$ .

Volatile in steam.

Acetyl: cryst.

Bamberger, Bokmann, 1118. 20,

### Naphthylenediamine.

*N,N*-Diphenyl: see Diphenylnaphtamine. di-

### Naphthylisobutane.

See *tert*.-Butylnaphthalene.

### Naphthylmethyl bromide.

See Bromomethyl-naphthalene.

### Naphthylmethyl chloride.

See Chloromethyl-naphthalene.

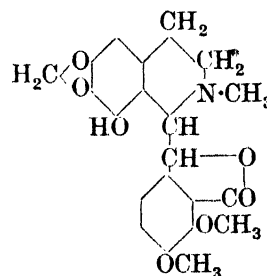
### Naphthylpropylene.

See Allylnaphthalene.

### 3- $\alpha$ -Naphthylvaleric Acid.

See 3-Methyl-3- $\alpha$ -naphthylbutyric Acid.

### Narcotoline



$C_{21}H_{21}O_7N$

MW, 399

Alkaloid from poppy, *Papaver somniferum*.

Rectangular rods from  $MeOH$ .Aq. M.p.  $202^\circ$ .

$[\alpha]_D^{20} - 189^\circ$  in  $CHCl_3$ ,  $+5.8^\circ$  in  $0.1N/HCl$ .

$CH_2N_2 \rightarrow$  narcotine.

*Bitartrate*: needles. Decomp. at about  $200^\circ$ .

Wrede, *Chem. Zentr.*, 1938, II, 863; 1937, I, 2611.

### Necholestene.

See  $\Delta^2$ -Cholestene.

### Neoline

$C_{24}H_{41}O_6N$

MW, 439

Alkaloid from *Aconitum napellus*. Prisms from  $Et_2O$ -pet. ether. M.p.  $153-4^\circ$ .  $[\alpha]_D^{20} +9.7^\circ$  in  $EtOH$ .

$B, HCl$ : cryst. from  $MeOH-Et_2O$ . Decomp. at  $178-80^\circ$ .

*B, HBr*: needles from MeOH-Et<sub>2</sub>O. M.p. 215° decomp.  $[\alpha]_D^{25} + 2.1^\circ$  in H<sub>2</sub>O.

Freudenberg, Rogers, *J. Am. Chem. Soc.*, 1937, **59**, 2572.

### Neopentyl Alcohol.

See *tert*.-Butylcarbinol.

### Neoprene.

See Chloroprene.

### Neoquassin

C<sub>22</sub>H<sub>30</sub>O<sub>6</sub> MW, 390

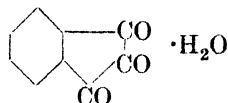
Occurs together with quassin in wood of *Quassia amara*. Prisms and plates from MeOH-Aq. M.p. 225.6°.  $[\alpha]_D^{20} + 46.6^\circ$  in CHCl<sub>3</sub>. Contains two CH<sub>3</sub>O groups and one active H atom. 3.5% HCl  $\rightarrow$  semidemethoxyquassin, C<sub>21</sub>H<sub>28</sub>O<sub>6</sub>, m.p. 213°.

Clark, *J. Am. Chem. Soc.*, 1937, **59**, 927, 2511; 1938, **60**, 1146.

### Niacin.

See Nicotinic Acid.

### Ninhydrin (Triketohydrindene hydrate)



C<sub>9</sub>H<sub>6</sub>O<sub>4</sub> MW, 178

Prisms from H<sub>2</sub>O. Anhyd. at 125–30°. M.p. 241–3° decomp. Alkalis  $\rightarrow$  yellow  $\rightarrow$  blue  $\rightarrow$  colourless sols. Reagent for the detection of uncombined amino and carboxyl groups in proteins, etc. Warm with amino acid  $\rightarrow$  blue col.

*Di-phenylhydrazone*: cryst. from EtOH. M.p. 180°.

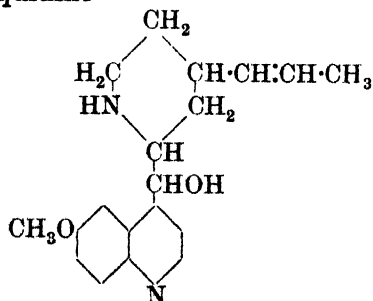
*Phenazine deriv.*: cryst. from EtOH. M.p. 219–20°.

Vanags, Lode, *Ber.*, 1938, **71**, 1267.

Teeters, Shriner, *J. Am. Chem. Soc.*, 1933, **55**, 3026.

Ruhemann, *J. Chem. Soc.*, 1910, **97**, 2025.

### Niquidine



C<sub>19</sub>H<sub>24</sub>O<sub>2</sub>N<sub>2</sub>

MW, 312

Needles from moist Et<sub>2</sub>O. M.p. 172°.  $[\alpha]_D^{18} + 301.5^\circ$  in 0.1N/H<sub>2</sub>SO<sub>4</sub>, + 186° in EtOH. H<sub>2</sub>O<sub>2</sub>  $\rightarrow$  quinic acid.

*B, HBr*: prisms. M.p. 217°.  $[\alpha]_D^{18} + 240^\circ$  in 0.1N/H<sub>2</sub>SO<sub>4</sub>.

*B, 2HBr*: prisms from H<sub>2</sub>O. M.p. 230° decomp.  $[\alpha]_D^{18} + 199^\circ$  in H<sub>2</sub>O.

*Hydrogen oxalyl*: needles. M.p. 215°.

Gibbs, Henry, *J. Chem. Soc.*, 1939, **240**, 1294.

### Nitrohydroxyethylbenzene.

See Nitromethylphenylcarbinol and Methyl-nitrophenyl-carbinol.

### Nitromethylbenzyl chloride.

See Nitroxylyl chloride.

**Nitromethylphenylcarbinol** (2-Nitro-1-phenylethyl alcohol,  $\beta$ -nitro- $\alpha$ -hydroxyethylbenzene)



C<sub>8</sub>H<sub>9</sub>O<sub>3</sub>N MW, 167

Yellow liq. B.p. 163–5°/15 mm. decomp. Mod. sol. H<sub>2</sub>O. CrO<sub>3</sub>  $\rightarrow$   $\omega$ -nitroacetophenone.

*Me ether*: C<sub>9</sub>H<sub>11</sub>O<sub>3</sub>N. MW, 181. B.p. 135–6°/12 mm.

*Et ether*: C<sub>10</sub>H<sub>13</sub>O<sub>3</sub>N. MW, 195. Yellowish oil. B.p. 136–7°/12 mm. Volatile in steam.

Holleman, *Rec. trav. chim.*, 1904, **23**, 299.

Rosenmund, *Ber.*, 1913, **46**, 1038.

Meisenheimer, Heim, *Ber.*, 1905, **38**, 469.

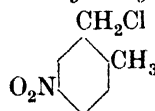
### Nitrophenylethyl Alcohol.

See Methylnitrophenylcarbinol.

### *p*-Nitrosobenzylaniline.

See Benzyl-*p*-nitrosoaniline.

**5-Nitro-*o*-xylyl chloride** ( $\omega$ -Chloro-5-nitro-*o*-xylene, 5-nitro-2-methylbenzyl chloride)

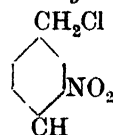


C<sub>8</sub>H<sub>8</sub>O<sub>2</sub>NCl MW, 185.5

Needles from MeOH. M.p. 50°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. Me<sub>2</sub>CO, ligroin. Irritates skin and mucous membrane. KMnO<sub>4</sub>  $\rightarrow$  4-nitrophthalic acid.

Stephen, Short, Gladding, *J. Chem. Soc.*, 1920, **117**, 526.

**3-Nitro-*p*-xylyl chloride** ( $\omega$ -Chloro-3-nitro-*p*-xylene, 3-nitro-4-methylbenzyl chloride)



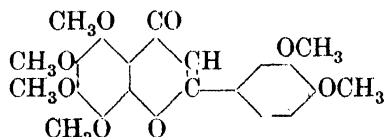
C<sub>8</sub>H<sub>8</sub>O<sub>2</sub>NC

MW, 185.5

Cryst. from MeOH. M.p. 45°. Sol. EtOH, Me<sub>2</sub>CO, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Irritates skin and mucous membrane. KMnO<sub>4</sub> → 2-nitroterephthalic acid.

Stephen, Short, Gladding, *J. Chem. Soc.*, 1920, 117, 525.

**Nobiletin** (5 : 6 : 7 : 8 : 3' : 4'-Hexamethoxy-flavone)



C<sub>21</sub>H<sub>22</sub>O<sub>8</sub> MW, 402

Constituent of peel of fruits of *Citrus nobilis*, Lour. Pale yellow cryst. from MeOH. M.p. 134°. Spar. sol. H<sub>2</sub>O, Et<sub>2</sub>O. EtOH-KOH → veratric acid + acetoveratrone.

Tseng, *J. Chem. Soc.*, 1938, 1003.

Robinson, Tseng, *ibid.*, 1004.

#### Nonadecanone-10.

See Caprinone.

#### Nonalupine

C<sub>15</sub>H<sub>24</sub>ON<sub>2</sub> MW, 248

Alkaloid from *Lupinus sericeus*, Pursh. Leaflets from methyl isobutyl ketone. M.p. 91.5-92.5°. When anhydrous, softens at 219°, m.p. 235°. B.p. 260-70°/18 mm. [α]<sub>D</sub><sup>25</sup> -21.3° in EtOH.

*Chloroaurate* : yellow needles from H<sub>2</sub>O. M.p. 177.5-178° decomp.

*Picrate* : cryst. from 50% EtOH. M.p. 185-6°.

Couch, *J. Am. Chem. Soc.*, 1940, 62, 554.

#### Nonamethylene bromide.

See 1 : 9-Dibromononane.

#### Nonamethylene chloride.

See 1 : 9-Dichlorononane.

#### Nonandione-2 : 8.

See 1 : 5-Diacetopentane.

#### 3-Nonene-1-carboxylic Acid.

See Obtusilic Acid.

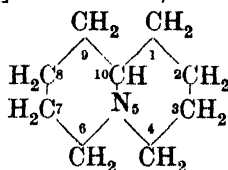
#### sec.-n-Nonylamine.

See 2-Amino-n-nonane.

#### Nonylone.

See Di-n-octyl Ketone.

**Norlupinane-A** (Octahydropyridocoline, bicyclo-[0 : 4 : 4]-aza-1-decane)



C<sub>9</sub>H<sub>17</sub>N

MW, 139

B.p. 74-6°/14 mm., 69-70°/11 mm.

*B, HBr* : prisms from CHCl<sub>3</sub>-AcOEt. M.p. 265-6°.

*B, H<sub>2</sub>AuCl<sub>4</sub>* : golden yellow leaflets or prisms from EtOH. M.p. 168°.

*Picrate* : yellow needles from EtOH. M.p. 194°.

*Picolonate* : yellow rods from AcOH. M.p. 245°.

*Methiodide* : plates from Me<sub>2</sub>CO. M.p. 333-5° decomp.

Clemo, Morgan, Raper, *J. Chem. Soc.*, 1935, 1743.

Clemo, Ramage, *J. Chem. Soc.*, 1931, 437, 3190.

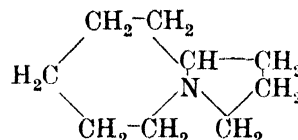
Clemo, Metcalfe, Raper, *J. Chem. Soc.*, 1936, 1430.

Prelog, Bozicevic, *Ber.*, 1939, 72, 1103.

Winterfield, Holschneider, *Ann.*, 1932, 499, 109.

Sugasawa, Lee, *J. Pharm. Soc. Japan*, 1939, 59, 326.

**Norlupinane-B** (Bicyclo-[0 : 3 : 5]-aza-1-decane)



C<sub>9</sub>H<sub>17</sub>N

MW, 139

B.p. 80°/14 mm.

*Picrate* : yellow needles from EtOH. M.p. 213-14° corr.

*Picolonate* : yellow tablets from EtOH. M.p. 191.5° corr.

*Methiodide* : needles from Me<sub>2</sub>CO. M.p. 282.5-283° corr.

Clemo, Ramage, *J. Chem. Soc.*, 1931, 437.

Prelog, Seiwerth, *Ber.*, 1939, 72, 1638.

#### Norvaline.

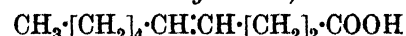
N-Phenyl : see 1-Anilinovaleric Acid.

## O

#### Obaculactone.

See Limonin.

**Obtusilic Acid** (3-Decenoic acid, 3-decylenic acid, 3-nonene-1-carboxylic acid)



C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>

MW, 170

Constituent of seed oil of *Lindera obtusiloba*.

B.p. 148-50°/13 mm. D<sub>4</sub><sup>20</sup> 0.9197. n<sub>D</sub><sup>20</sup> 1.4497.

p-Bromophenacyl ester : m.p. 43.3°.

Komori, Ueno, *Bull. Chem. Soc. Japan*, 1937, 12, 226, 433.

**Ochotensine** $C_{21}H_{21}O_4N$  MW, 351

Alkaloid of *Corydalis ochotensis*, Turcz. M.p. 252°.  $[\alpha]_D^{25} + 63.9^\circ$  in 0.1N/HCl,  $[\alpha]_D^{25} + 51.7^\circ$  in  $CHCl_3$ . Spar. sol.  $Et_2O$ . Phenolic.

Manske, *Chem. Abstracts*, 1940, **34**, 4070.

**Ochrobirine** $C_{20}H_{19}O_6N$  MW, 369

Alkaloid of *Corydalis lutea*. M.p. 138-9°.  $[\alpha]_D^{21} + 35.9^\circ$  in  $CHCl_3$ .

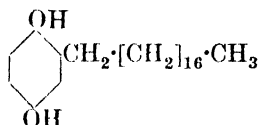
Acetyl deriv.: m.p. 177°.

Manske, *Chem. Abstracts*, 1939, **33**, 6323.

**9 : 12-Octadecadienol-1.**

See Linoleyl Alcohol.

**Octadecylhydroquinone** (*Octadecylquinol*, 2 : 5-dihydroxyoctadecylbenzene)

 $C_{24}H_{42}O_2$  MW, 362

Cryst. from pet. ether. M.p. 100.5°.  $Ag_2O \rightarrow$  octadecylbenzoquinone. M.p. 76°.

*Di-Me ether*: 2 : 5-dimethoxyoctadecylbenzene. B.p. 188°/0.2 mm.

*Di-Et ether*: 2 : 5-diethoxyoctadecylbenzene. B.p. 201°/0.06 mm.

Cook, Heilbron, Lewis, *J. Chem. Soc.*, 1942, 660.

**Octahydropyridocoline.**

See Norlupinane-A.

**Octamethylene bromide.**

See 1 : 8-Dibromo-octane.

**Octamethylene chloride.**

See 1 : 8-Dichloro-octane.

**5-Octanolone-4.**

See Butyrolin.

**2 : 4 : 6-Octatrienal** $C_8H_{10}O$  MW, 122

Needles from pet. ether. M.p. 55°. B.p. 57-68°/0.5 mm. Sol. org. solvents. Absorption maximum at 3160 Å in EtOH.

*Oxime*: yellow. M.p. 186-7° decomp.

*Hydrazone*: yellow needles from EtOH. Sinters at 153-4°.

*Azine*: orange cryst. from butyl or amyl alcohol. Decomp. at 220-5°.

Fischer, Hultsch, Flaig, *Ber.*, 1937, **70**, 370.

Kuhn, Hoffer, *Ber.*, 1930, **63**, 2164; 1931, **64**, 1977.

Kuhn, *Angew. Chem.*, 1937, **50**, 703.

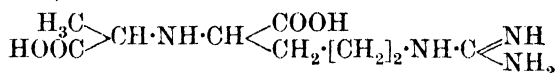
Gödde, *Chem. Abstracts*, 1940, **34**, 1970.

**2 : 4 : 6-Octatrienol** $C_8H_{12}O$  MW, 124

Needles. M.p. 99.5-100.5°. B.p. 95°/12 mm.

Reichstein, Ammann, Trivelli, *Helv. Chim. Acta*, 1932, **15**, 261, 502.

**Octopin** (*Pectenin*, 1-arginine-1- $\alpha$ -propionic acid)

 $C_9H_{18}O_4N_4$  MW, 246

Isolated from aq. extracts of tentacle muscles of *Loligo pealii* and *Octopus vulgaris*, the adductor muscles of the scallop *Pecten magellanicus* and the muscles of the octopod *Eledone moschata*. Needles from  $H_2O$  or EtOH.Aq. M.p. 281-2° (261-4°). Odourless and tasteless. Readily sol. hot  $H_2O$ . Spar. sol. MeOH, EtOH,  $Et_2O$ .  $[\alpha]_D^{27} + 20.94^\circ$  in  $H_2O$ .

*Cu salt*: m.p. 223-7° decomp.

*Ni salt*: m.p. above 290°.

*Picrate*: m.p. 226-30° corr. decomp.

*Picrolonate*: m.p. 237-9° corr. decomp.

Akashi, *Chem. Abstracts*, 1937, **31**, 5766.

Knoop, Martius, *Z. physiol. Chem.*, 1938, **254**, 1; 1939, **258**, 238.

Irvin, Wilson, *J. Biol. Chem.*, 1939, **127**, 555, 565, 575.

Karrer, Koenig, Legler, *Helv. Chim. Acta*, 1941, **24**, 127.

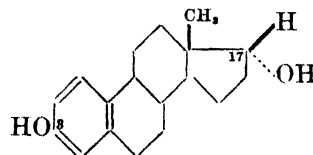
**sec.-n-Octylamine.**

See 2-Amino-n-octane.

**Oenanthylidene.**

See 1-Heptene.

**$\alpha$ -Oestradiol** (*trans-Oestradiol*, *trans-di-hydro- $\alpha$ -estrone*)

 $C_{18}H_{24}O_2$  MW, 272

Isolated from ovaries and pregnancy urine. Leaflets or needles from EtOH.Aq. Cryst. from  $Me_2CO$ . M.p. 174°.  $[\alpha]_D^{25} + 78^\circ$  in EtOH. Digitonide is insol. 80% EtOH. The most potent of the natural oestrogens.

3-Me ether: needles from MeOH.Aq. M.p. 97-8°.

3-Acetyl: m.p. 137°.

17-Acetyl: m.p. 215-17°.

Diacetyl: leaflets from MeOH.Aq. M.p. 125-6°.

3-Propionyl: m.p. 125°.

17-Propionyl: m.p. 199-200°.

Dipropionyl: m.p. 104-5°.

17-n-Butyryl: m.p. 166-7°.

Di-n-butyryl: m.p. 64-5°.

3-Trimethylacetyl: needles from MeOH.Aq. M.p. 178-80°.

Di-trimethylacetyl: needles from Me<sub>2</sub>CO-MeOH. M.p. 174-6°.

3-Palmityl: m.p. 69-71°.

3-Benzoyl: needles from EtOH.Aq. M.p. 192-3°.

3:17-Dibenzoyl: leaflets from EtOH. M.p. 168-9°.

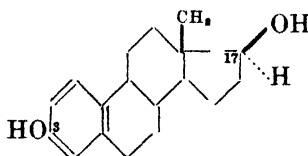
Butenandt, *Z. physiol. Chem.*, 1937, **248**, 129.

Doisy, *J. Biol. Chem.*, 1936, **115**, 435.

Wintersteiner, *J. Biol. Chem.*, 1937, **118**, 789; *J. Am. Chem. Soc.*, 1937, **59**, 765.

Marker, *J. Am. Chem. Soc.*, 1938, **60**, 2927.

**$\beta$ -Oestradiol** (cis-Oestradiol, cis-dihydro-*œstrone*)



C<sub>18</sub>H<sub>24</sub>O<sub>2</sub> MW, 272

Isolated from pregnancy urine. Needles from EtOH.Aq. Cryst. from Me<sub>2</sub>CO. M.p. 223°. [ $\alpha$ ]<sub>D</sub><sup>25</sup> +56.7° in EtOH. Much less potent than the  $\alpha$ -isomer.

3-Me ether: leaflets from MeOH. M.p. 109-10°.

3:17-Diacetyl: needles from EtOH.Aq. M.p. 139-40°.

3-Benzoyl: leaflets from MeOH.Aq. M.p. 156-7°.

Butenandt, *Z. physiol. Chem.*, 1937, **248**, 129.

Wintersteiner, *J. Biol. Chem.*, 1938, **122**, 303; *J. Am. Chem. Soc.*, 1937, **59**, 765.

Marker, *J. Am. Chem. Soc.*, 1938, **60**, 2927.

#### Oleic Acid.

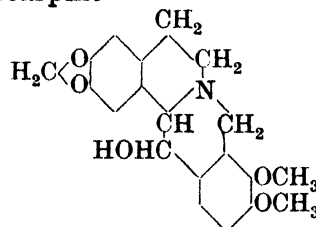
Dibromide: see 8:9-Dibromostearic Acid.

Dichloride: see 8:9-Dichlorostearic Acid.

#### Oöcyan.

See Biliverdin..

#### Ophiocarpine



C<sub>20</sub>H<sub>21</sub>O<sub>5</sub>N

MW, 355

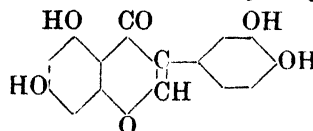
Principal alkaloidal constituent (0.25%) of *Corydalis ophiocarpa*. M.p. 188°. [ $\alpha$ ]<sub>D</sub><sup>24</sup> -284° in CHCl<sub>3</sub>.

Manske, *Chem. Abstracts*, 1939, **33**, 6321.

#### Orcacetophenone.

See 4:6-Dihydroxy-2-methylacetophenone.

**Orobol** (5:7:3':4'-Tetrahydroxyisoflavone)



C<sub>15</sub>H<sub>10</sub>O<sub>6</sub>

MW, 286

Occurs as glucoside, oroboside, in *Orobis tuberosus*, L. Pale yellow needles from AcOH. M.p. 270.5°. 30% KOH  $\rightarrow$  phloroglucinol + homoprotocatechuic acid.

Tetra-acetyl: cryst. from AcOH. M.p. 210-12°.

Bridel, Charaux, *Compt. rend.*, 1930, **190**, 387; *Bull. soc. chim. biol.*, 1930, **12**, 615.

Charaux, Rabaté, *Bull. soc. chim. biol.*, 1939, **21**, 1330.

#### Oroboside

C<sub>21</sub>H<sub>20</sub>O<sub>11</sub>

MW, 448

Occurs in *Orobis tuberosus*, L. Yellow prisms from 40% EtOH.Aq. M.p. 220-1°. [ $\alpha$ ]<sub>D</sub> -61.3° in Py. Acid hyd.  $\rightarrow$  orobol + glucose.

Bridel, Charaux, *Compt. rend.*, 1930, **190**, 387; *Bull. soc. chim. biol.*, 1930, **12**, 615.

#### p-Orsellinaldehyde.

See Atranol.

#### Oryzanin.

See Aneurin.

#### Osajin

C<sub>25</sub>H<sub>24</sub>O<sub>5</sub>

MW, 404

Isolated from fruit of osage orange tree (*Maclura pomifera*, Raf.). Lemon yellow cryst.

from xylene or 95% EtOH. M.p. 189° (193° corr.). Very sol.  $\text{CHCl}_3$ ,  $\text{Et}_2\text{O}$ ,  $\text{Me}_2\text{CO}$ , Py. Mod. sol.  $\text{C}_6\text{H}_6$ , warm  $\text{CCl}_4$ . Prac. insol.  $\text{H}_2\text{O}$ , pet. ether. Reduces Fehling's, and Tollen's in Py. Alc. sol. + aq.  $\text{FeCl}_3 \rightarrow$  dark green col.  $\rightarrow$  reddish violet on addn. of a few drops of  $\text{NH}_4\text{OH}$ .

*Monoacetyl deriv.*: yellow cryst. from EtOH. M.p. 159°.

*Diacetyl deriv.*: cryst. from EtOH.Aq. Two forms. M.ps. 162° and 152°.

*Mono-p-toluenesulphonyl*: yellow plates from EtOH. M.p. 152°.

*Me ether*:  $\text{C}_{26}\text{H}_{26}\text{O}_5$ . MW, 418. Yellow needles from 95% EtOH. M.p. 134–5°. *Monoacetyl*: prisms from 95% EtOH. M.p. 140–140.5°.

*Di-Me ether*:  $\text{C}_{27}\text{H}_{28}\text{O}_5$ . MW, 432. Needles from 95% EtOH. M.p. 118.5°.

Wolfrom, Gregory, *J. Am. Chem. Soc.*, 1940, **62**, 651.

Wolfrom, Benton, Gregory, Hess, Mahan, Morgan, *J. Am. Chem. Soc.*, 1939, **61**, 2833.

Walter, Wolfrom, Hess, *J. Am. Chem. Soc.*, 1938, **60**, 575.

## P

### Palustrin

$\text{C}_{12}\text{H}_{24}\text{O}_2\text{N}_2$  MW, 228

Alkaloid isolated from swamp horsetail (*Equisetum palustre*). Pale yellow oil with amine-like odour. B.p. 205–10°/0.1 mm.

*B,HCl*: cubes. M.p. 181°.

Glet, Gutschmidt, Glet, *Z. physiol. Chem.*, 1936, **244**, 229.

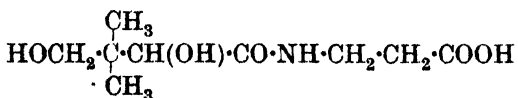
### Pamaquin.

See Plasmoquin.

### Pantocaine.

See Decicaine.

### Pantothenic Acid



$\text{C}_9\text{H}_{17}\text{O}_5\text{N}$  MW, 219

*d.*

Growth-promoting and antidermatitic factor ("Filtrate factor") of vitamin B complex, present in living cells particularly of the liver. Viscous oil.  $[\alpha]_D^{25} + 37.5^\circ$  in  $\text{H}_2\text{O}$ . Acid or alk. hyd.  $\rightarrow$   $\beta$ -alanine + *l*- $\alpha$ -hydroxy- $\beta\beta$ -di-

methyl- $\gamma$ -butyrolactone (or salt of corresponding acid).

*Na salt*: cryst. from EtOH. M.p. 122–4° (121–2°).  $[\alpha]_D^{19.5} + 29.5^\circ$  in  $\text{H}_2\text{O}$ ,  $[\alpha]_D^{25} + 27.04^\circ$  in  $\text{H}_2\text{O}$ .

*Ca salt*: cryst. from MeOH. M.p. 195–6°.  $[\alpha]_D^{25} + 28.2^\circ$  in  $\text{H}_2\text{O}$ . Anisotropic.

*Benzylthiuronium salt*: m.p. 148–9°.

*Cinchonidine salt*: m.p. 178–9° (176–7°).  $[\alpha]_D^{25.4} - 60.6^\circ$  in MeOH,  $[\alpha]_D^{18} - 62.8^\circ$  in  $\text{H}_2\text{O}$ .

*Quinine salt*: m.p. 136–7°.  $[\alpha]_D^{19} - 95^\circ$  in  $\text{H}_2\text{O}$ .

*Me ester*:  $\text{C}_{10}\text{H}_{19}\text{O}_5\text{N}$ . MW, 233. Oil.  $[\alpha]_D^{18} + 37.1^\circ$  in  $\text{Me}_2\text{CO}$ .

*Et ester*:  $\text{C}_{11}\text{H}_{21}\text{O}_5\text{N}$ . MW, 247. Oil.  $[\alpha]_D^{18} + 36.8^\circ$  in EtOH.

*l.*

Little or no biological activity (conflicting results). Viscous oil.  $[\alpha]_D^{21} - 26.7^\circ$  in  $\text{H}_2\text{O}$ ,  $-56.3^\circ$  in MeOH.

*Na salt*: m.p. 120–2°.  $[\alpha]_D^{18} - 27.4^\circ$  in  $\text{H}_2\text{O}$ .

*Ca salt*: cryst. from MeOH. M.p. 187.5–189°.  $[\alpha]_D^{26} - 27.8^\circ$  in  $\text{H}_2\text{O}$ .

*Benzylthiuronium salt*: m.p. 150–1° (145–6°).

*Quinine salt*: needles from  $\text{Me}_2\text{CO}$ -MeOH. M.p. 183.0° (167–8°).  $[\alpha]_D^{19} - 121^\circ$  in  $\text{H}_2\text{O}$ .

*Et ester*: oil.  $[\alpha]_D^{19} - 37.3^\circ$  in EtOH.

*dl.*

Viscous oil. Possesses half biological activity of *d*-form.

*Benzylthiuronium salt*: needles from  $\text{Me}_2\text{CO}$ . M.p. 135–6°.

Woolley *et al.*, *J. Biol. Chem.*, 1939, **129**, 673.

Williams *et al.*, *J. Am. Chem. Soc.*, 1940, **62**, 1776, 1784.

Stiller *et al.*, *J. Am. Chem. Soc.*, 1940, **62**, 1779, 1785; 1941, **63**, 1237, 2846.

Woolley, *J. Am. Chem. Soc.*, 1940, **62**, 2251.

Reichstein *et al.*, *Helv. Chim. Acta*, 1940, **23**, 650, 1276; 1941, **24**, 185.

Kuhn *et al.*, *Ber.*, 1940, **73**, 971, 1134; 1941, **74**, 218.

Parke, Lawson, *J. Am. Chem. Soc.*, 1941, **63**, 2869.

Harris, Boyack, Folkers, *J. Am. Chem. Soc.*, 1941, **63**, 2662.

### Paracasein.

See Caseinogen.

### Parachloralose.

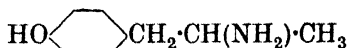
See  $\beta$ -Glucochloralose.

### Paracyanoformic Acid.

See Triazine-tricarboxylic Acid.



**Paredrine** (1 - p - Hydroxyphenylisopropylamine, p-β-aminopropylphenol)



$C_9H_{13}ON$

MW, 151

Exerts marked pressor activity similar to that of benzedrine.

*B.HCl*: cryst. from EtOH-Et<sub>2</sub>O. M.p. 171-2°. Sol. H<sub>2</sub>O, Et<sub>2</sub>O.

Alles, *J. Am. Chem. Soc.*, 1932, **54**, 271.

Woodruff, Conger, *J. Am. Chem. Soc.*, 1938, **60**, 465.

Iglauer, Altschule, *American Journal of Medical Sciences*, 1940, **199**, 359, (*Chem. Abstracts*, 1940, **34**, 3821).

### Parinaric Acid



$C_{18}H_{28}O_2$

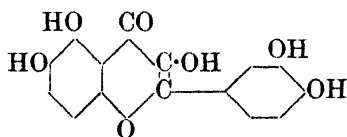
MW, 276

Acid of the kernel fat of *Parinarium laurinum*. Faintly yellow plates from pet. ether. M.p. 85-6°. Isomerised by I or light to acid, m.p. 95-6°. Absorption maxima: 3200, 3070 and 2920 Å. in EtOH.

Farmer, Sunderland, *J. Chem. Soc.*, 1935, 759.

Kaufmann, Baltes, Funke, *Chem. Zentr.*, 1938, II, 4143.

### Patuletin



Probable structure

$C_{15}H_{10}O_7$

MW, 302

Colouring matter of flowers of *Tagetes patula*. Pale yellow needles from EtOH.Aq. M.p. 262-4°. Alk. ox. → protocatechuic acid.

*Penta-acetyl*: cryst. from AcOH.Aq. M.p. 170-2°.

*Penta-Me ether*:  $C_{20}H_{20}O_7$ . MW, 372. Cryst. from AcOH.Aq. M.p. 158-9° after sintering at 143°.

Rao, Seshadri, *Proceedings of the Indian Academy of Sciences*, 1941, **14**, 643, (*Brit. Chem. Abstracts*, 1942, A II, 202).

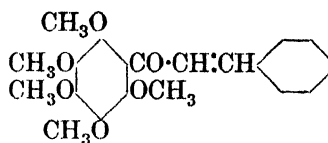
### Pectenin.

See Octopin.

### Pectenine.

See Carnegine.

### Pedicellin



$C_{20}H_{22}O_6$

MW, 358

Isolated from dried leaves of *Didymocarpus pedicellata*. Cryst. from Et<sub>2</sub>O. M.p. 93°. HNO<sub>3</sub> → methylpedicinin ( $C_{17}H_{14}O_6$ ), m.p. 110°.

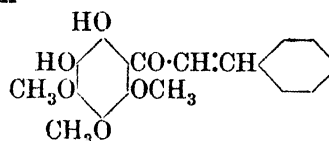
*Phenylhydrazone*: rods from AcOEt. M.p. 133-5°.

Sharma, Siddiqui, *J. Indian Chem. Soc.*, 1939, **16**, 1.

Bose, Dutt, *J. Indian Chem. Soc.*, 1940, **17**, 499.

Baker, *J. Chem. Soc.*, 1941, 662.

### Pedicin



Suggested structure

$C_{18}H_{18}O_6$

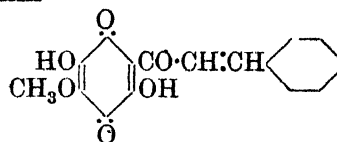
MW, 330

Isolated from coarsely powdered leaves of *Didymocarpus pedicellata*. Orange red plates. M.p. 145°. Br in CHCl<sub>3</sub> or KMnO<sub>4</sub> → pedicin. Methylation → pedicellin.

*Dibenzoyl*: rods from C<sub>6</sub>H<sub>6</sub>. M.p. 181-3°. *Phenylhydrazone*: needles from EtOH. M.p. 165-7°.

Siddiqui *et al.*, *J. Indian Chem. Soc.*, 1937, **14**, 703; 1939, **16**, 1, 423.

### Pedicinin



Probable structure

$C_{18}H_{12}O_6$

MW, 300

Isolated from dried leaves of *Didymocarpus pedicellata*. Red rods or needles from C<sub>6</sub>H<sub>6</sub>. M.p. 203°. Alkalis → benzaldehyde.

*Monoacetyl*: prisms from AcOEt. M.p. 175°.

Siddiqui, *J. Indian Chem. Soc.*, 1937, **14**, 703.

Bose, Dutt, *J. Indian Chem. Soc.*, 1940, **17**, 499.

**Peiminine** $C_{26}H_{43}O_3N$ 

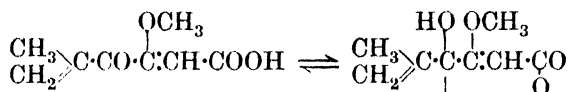
MW, 417

Alkaloid of Chinese drug *Pei-Mu* from corms of *Fritillaria Roylei*, Stuart. Needles from EtOH-pet. ether. Sinters at 140°, melts at 147-8°, solidifies at 157°, finally melts at 212-13°.  $[\alpha]_D^{18} - 65.8^\circ$ .

*Hydrochloride*: m.p. 292°.

*Hydrobromide*: m.p. 295°.

Chi, Kao, Chang, *J. Am. Chem. Soc.*, 1940, **62**, 2896; 1936, **58**, 1306.

**Penicillic Acid** $C_8H_{10}O_4$ 

MW, 170

Metabolic product of *Penicillium puberulum* Bainier, and *Penicillium cyclopium* Westling. Rhombic or hexagonal plates + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 64-5°, anhyd. 87°. Sol. to 2% in cold H<sub>2</sub>O. Readily sol. hot H<sub>2</sub>O, EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Insol. pet. ether. NH<sub>4</sub>OH → reddish purple col.

*Acetyl*: prisms from pet. ether. M.p. 72°.

*Dimedone deriv.*: C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>, C<sub>8</sub>H<sub>12</sub>O<sub>2</sub>. Cryst. from EtOH.Aq. M.p. 201.3°.

*Dihydro-comp.*: needles + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 62-4°, anhyd. 83-5°.

*Dibromide*: needles from CCl<sub>4</sub>. M.p. 154-5°.

Birkinshaw, Oxford, Raistrick, *Biochem. J.*, 1936, **30**, 394.

**Pentadecanol-8.**

See Di-*n*-heptylcarbinol.

**Pentadecanone-8.**

See Caprylone.

**Pentahydroxy-1-amino-hexane.**

See Glucamine.

**Pentahydroxycyclohexane.**

See Viburnitol.

**3:5:7:3':4'-Pentahydroxyflavan.**

See Catechin.

**3:5:7:8:4'-Pentahydroxyflavone.**

See Herbacetin.

**Pentahydroxyhexylamine.**

See Glucamine.

**3:5:6:7:8 - Pentahydroxy - 2 - ethyl - 1 : 4 - naphthoquinone.**

See Echinochrome A.

**Pentamethylene-tetrazole.**

See Cardiazole.

**Pentane-3-carboxylic Acid.**

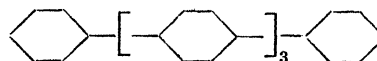
See Diethylacetic Acid.

**Pentane-1 : 3-dicarboxylic Acid.**

See 1-Ethylglutaric Acid.

**Pentanolamine.**

See Aminopentanol.

**p-Pentaphenyl (p-Quinquiphenyl)** $C_{30}H_{22}$ 

MW, 382

Needles from quinoline. M.p. 388-5°. Sublimes.

Gerngross, Dunkel, *Ber.*, 1924, **57**, 739.

Bursch, Weber, *J. prakt. Chem.*, 1936, **146**, 29.

Müller, Töpel, *Ber.*, 1939, **72**, 290.

**3-Pentenic Acid.**

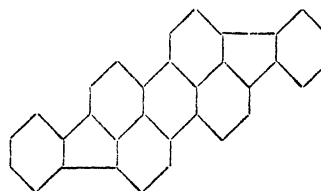
See Allylacetic Acid.

**1-Pentitol-3.**

See Ethylethynylcarbinol.

**Perbenzoic Acid.**

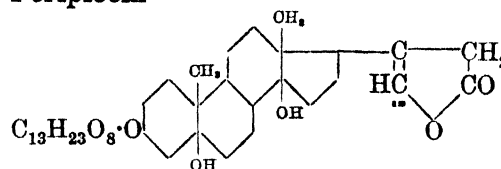
*Benzoyl*: see Benzoyl peroxide.

**Periflanthene** $C_{32}H_{16}$ 

MW, 400

Dark red solid. M.p. above 360°. Spar. sol. chlorobenzene, bromobenzene, phenol. Insol. Et<sub>2</sub>O, xylene. Sublimes. Very stable and very resistant to oxidising agents. Sol. conc. H<sub>2</sub>SO<sub>4</sub> at 100° with blue col.

Braun, Manz, *Ber.*, 1937, **70**, 1603.

**Periplocin**

Suggested structure

 $C_{36}H_{56}O_{13}$ 

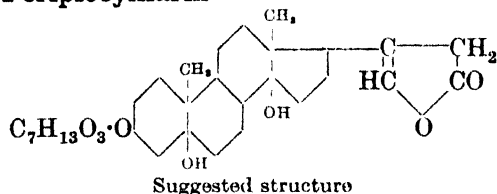
MW, 696

Cardiac glycoside from *Periploca graeca*, L. Needles from H<sub>2</sub>O. M.p. 224° decomp. Sol. EtOH. Spar. sol. H<sub>2</sub>O. Prac. insol. Et<sub>2</sub>O, CHCl<sub>3</sub>.  $[\alpha]_D^{20} + 22.9^\circ$  in MeOH. Hyd. → periplogenin + periplobiose. Enzyme hyd. → periplocymarin.

*Tetra-acetyl*: prisms from EtOH. M.p. 195°.

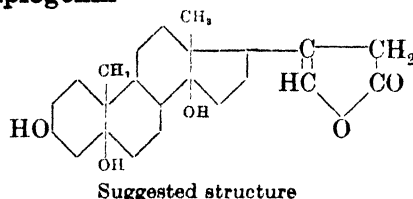
Sol. EtOH,  $\text{CHCl}_3$ . Spar. sol.  $\text{H}_2\text{O}$ .  $[\alpha]_D^{20} + 20.0^\circ$  in EtOH.

Lehmann, *Arch. Pharm.*, 1897, **235**, 157.  
Jacobs, Hoffmann, *J. Biol. Chem.*, 1928, **79**, 519.  
Stoll, Renz, *Helv. Chim. Acta*, 1939, **22**, 1193.

**Periplocymarin**

$\text{C}_{30}\text{H}_{46}\text{O}_8$  MW, 534  
Prisms from MeOH.Aq. M.p.  $148-51^\circ$ .  $[\alpha]_D^{20} + 27.6^\circ$  in MeOH.  $\text{HCl} \longrightarrow$  periplogenin.

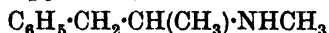
Jacobs, Hoffmann, *J. Biol. Chem.*, 1928, **79**, 519.  
Stoll, Renz, *Helv. Chim. Acta*, 1939, **22**, 1207.  
Solacolu, Herrmann, *Compt. rend. soc. biol.*, 1934, **117**, 1138.

**Periplogenin**

$\text{C}_{23}\text{H}_{34}\text{O}_5$  MW, 390  
Aglucone of periplocin. Prisms from MeOH.Aq. M.p.  $232^\circ$ .  $[\alpha]_D^{27} + 31.5^\circ$  in EtOH.

Lehmann, *Arch. Pharm.*, 1897, **235**, 157.  
Jacobs, Elderfield, *J. Biol. Chem.*, 1935, **108**, 497.  
Kon, *J. Soc. Chem. Ind.*, 1934, **53**, 593.  
Stoll, Renz, *Helv. Chim. Acta*, 1939, **22**, 1193.

**Pervitin** (2-Methylamino-1-phenylpropane, N:  $\alpha$ -dimethylphenylethylamine, N-methyl-1-phenylisopropylamine)



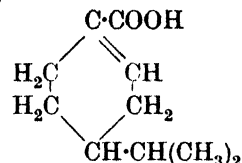
$\text{C}_{10}\text{H}_{15}\text{N}$  MW, 149

B.p.  $78-80^\circ/6$  mm. Exhibits analeptic action. Excitatory and peripheral action similar to that of benzedrine.

B, HCl: cryst. from EtOH-Et<sub>2</sub>O. M.p.  $135-6^\circ$ .

Woodruff, Lambooy, Burt, *J. Am. Chem. Soc.*, 1940, **62**, 922.

**Phellandric Acid** (3:4:5:6-Tetrahydro-p-isopropylbenzoic acid, 3:4:5:6-tetrahydro-cuminic acid)



$\text{C}_{10}\text{H}_{16}\text{O}_2$  MW, 168

dl-.

Cryst. from EtOH.Aq. M.p.  $144-5^\circ$ .  $[\alpha]_D^{20} + 112.8^\circ$  in MeOH.

p-Chlorophenacyl ester: needles from EtOH.Aq. M.p.  $78-78.5^\circ$ .  $[\alpha]_D^{20} + 71^\circ$  in  $\text{CHCl}_3$ .

p-Bromophenacyl ester: needles from EtOH.Aq. M.p.  $86^\circ$ .  $[\alpha]_D^{20} + 68.1^\circ$  in  $\text{CHCl}_3$ .

l-.

M.p.  $144-5^\circ$ .  $[\alpha]_D^{20} - 112.6^\circ$  in MeOH.

p-Chlorophenacyl ester: needles from EtOH.Aq. M.p.  $78-78.5^\circ$ .  $[\alpha]_D^{20} - 57^\circ$  in  $\text{CHCl}_3$ .

p-Bromophenacyl ester: needles from EtOH.Aq. M.p.  $86^\circ$ .  $[\alpha]_D^{20} - 52.2^\circ$  in  $\text{CHCl}_3$ .

p-Nitrobenzyl ester: pale yellow needles from MeOH. M.p.  $56-7^\circ$ .

dl-.

Cryst. from MeOH.Aq. M.p.  $143-4^\circ$ .

p-Bromophenacyl ester: m.p.  $86-86.5^\circ$ .

Cooke, Macbeth, Swanson, *J. Chem. Soc.*, 1940, 808.

**Phemitone.**

See Prominal.

**Phenacyl Aldehyde.**

See Benzoylactaldehyde.

**Phenacylformaldehyde.**

See Benzoylactaldehyde.

**1-Phenacylpropionic Acid.**

See 2-Benzoylisobutyric Acid.

**Phenanthracridine.**

See 1:2:3:4-Dibenzacridine.

"Phenarsazine chloride."

See 10-Chloro-5:10-dihydrophenarsazine.

**Phenmorpholone.**

See under 2-Aminophenoxyacetic Acid.

**Phenothiazine.**

See Thiodiphenylamine.

**p-Phenylacetophenone.**

See 4-Acetodiphenyl.

**Phenylacetylene.**

Dibromide: see  $\alpha$ : $\beta$ -Dibromostyrene.

Dichloride: see  $\alpha$ : $\beta$ -Dichlorostyrene.

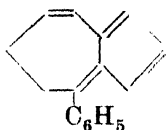
**Phenylallylamine.**

See Cinnamylamine and under Aniline.

**Phenyl aminotolyl Ketone.**

See Amino-methylbenzophenone.

## 4-Phenylazulene

 $C_{16}H_{12}$ 

MW, 204

Blue oil.

*Picrate*: black needles from EtOH. M.p. 80–1°.

sym.-*Trinitrobenzene add. comp.*: black needles from EtOH. M.p. 86–7°.

St. Pfau, Plattner, *Helv. Chim. Acta*, 1936, 19, 878.

**Phenylbenzylcarbinol** (1 : 2-Diphenylethyl alcohol,  $\alpha$ -hydroxydiphenylethane)

 $C_{14}H_{14}O$ 

MW, 198

dl-.

Needles from pet. ether-benzene. M.p. 67°.

B.p. 177°/15 mm.

*Acetyl*: b.p. 202–5°/10 mm.

*Benzoyl*: needles. M.p. 70°.

*Hydrogen phthaloyl*: needles from pet. ether-Et<sub>2</sub>O. M.p. 131°.

*Phenylurethane*: m.p. 94–5°.

d-.

Needles from Et<sub>2</sub>O-pet. ether. M.p. 67°.  $D_4^{20}$  1.0358.  $[\alpha]_D^{25}$  – 8.5° in CHCl<sub>3</sub>, + 53° in EtOH.

*Hydrogen phthaloyl*: needles from Et<sub>2</sub>O-pet. ether. M.p. 131°.  $[\alpha]_D^{25}$  + 33° in EtOH. *Quinine salt*: needles from EtOH. M.p. 205°.

l-.

Needles from Et<sub>2</sub>O-pet. ether. M.p. 67°.

*Acetyl*: b.p. 182°/15 mm.  $D_4^{17}$  1.0831.  $[\alpha]_D^{17}$  + 23.5°.

*Hydrogen phthaloyl*: needles from Et<sub>2</sub>O-pet. ether. M.p. 131°.  $[\alpha]_{5461}^{25}$  – 39° in EtOH. *Cinchonine salt*: cryst. from MeOH-Me<sub>2</sub>CO. M.p. 110°.

Gerrard, Kenyon, *J. Chem. Soc.*, 1928, 2564.

**Phenylbenzylideneacetone.**

See Benzyl styryl Ketone.

**Phenylbenzylidenebutiric Acid.**

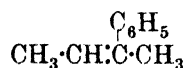
See Diphenylbutylene-carboxylic Acid.

**Phenylbenzylsuccinic Acid.**

See 1 : 3-Diphenylpropane-1 : 2-dicarboxylic Acid.

**Phenylbenzylvinylacetic Acid.**

See 1 : 4-Diphenyl-2-butylene-1-carboxylic Acid.

2-Phenyl-2-butylene ( $\alpha\beta$ -Dimethylstyrene) $C_{10}H_{12}$ 

MW, 132

B.p. 187–9°, 80–1°/20 mm., 73°/14 mm.  $D_4^{25}$  0.8911,  $D_4^{20}$  0.9088.  $n_D^{20}$  1.5339.

Haller, Bauer, *Ann. chim.*, 1918, 9, 12.

Klages, *Ber.*, 1902, 35, 2641, 3508.

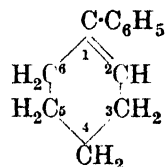
**Phenyl-chlorobenzylamine.**

See under Chlorobenzylamine.

**Phenyl chlorobenzoyl Ketone.**

See Chlorodeoxybenzoin.

**1-Phenylcyclohexene** (3 : 4 : 5 : 6-Tetrahydrodiphenyl)

 $C_{12}H_{14}$ 

MW, 158

Mobile liq. B.p. 250°, 133°/20 mm., 125–6°/14 mm.  $D_4^{14.9}$  0.9930,  $D_4^{20}$  0.9931,  $D_4^{25.2}$  0.9871.  $n_D^{20}$  1.5718 (1.5670),  $n_D^{25.2}$  1.5669.  $KMnO_4 \rightarrow$  4-benzoylvaleric acid.

Price, Karabinos, *J. Am. Chem. Soc.*, 1940, 62, 1160.

Sabatier, Mailhe, *Compt. rend.*, 1904, 138, 1323.

**3-Phenylcyclohexene** (1 : 2 : 3 : 4-Tetrahydrodiphenyl).

B.p. 115–17°/16 mm.  $D_4^{20}$  0.9800.  $n_D^{20}$  1.5530. Reflux with 5% HNO<sub>3</sub>  $\rightarrow$  2-phenyladipic acid.

Price, Karabinos, *J. Am. Chem. Soc.*, 1940, 62, 1160.

**4-Phenylcyclohexene** (1 : 2 : 5 : 6-Tetrahydrodiphenyl).

B.p. 88–90°/16 mm.  $D_4^{20}$  0.9715.  $n_D^{20}$  1.5420.  $KMnO_4 \rightarrow$  2-phenyladipic acid.

Price, Karabinos, *J. Am. Chem. Soc.*, 1940, 62, 2243.

**Phenyldichloroacetic Acid.**

See  $\alpha$  :  $\alpha$ -Dichlorophenylacetic Acid.

**Phenyl 3 : 4-dihydroxystyryl Ketone.**

See 3 : 4-Dihydroxychalkone.

**Phenyl dihydroxytolyl Ketone.**

See Dihydroxy-methylbenzophenone.

**N-Phenyl-dinitrobenzylamine.**

See Dinitrobenzylaniline.

**Phenyldodecane.**

See Dodecylbenzene.

**Phenylenediamine.**

*Carbathoxyl*: see Aminophenylurethane.

***o*-Phenyleneurea.**

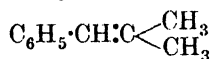
See Benziminazolone.

**Phenylethanolamine.**

See Hydroxyaminoethylbenzene.

**Phenyliminodeoxybenzoin.**

See under Benzil.

**1-Phenylisobutylene** ( $\beta\beta$ -Dimethylstyrene)

$\text{C}_{10}\text{H}_{12}$  MW, 132

M.p. — 52°. B.p. 183–5°, 76–7°/11 mm.  $D_4^{20}$  0.8980.  $n_D^{20}$  1.5273.

*Nitrosite*: needles from  $\text{C}_6\text{H}_6$ . M.p. 122° decomp. (154° decomp.).

Lévy, Tabart, *Bull. soc. chim.*, 1931, 49, 1776.

**Phenylisophthalic Acid.**

See Diphenyl-dicarboxylic Acid.

**Phenylisopropylamine.**

Benzedrine is 2-amino-1-phenylpropane. See p. 426, where the compound is incorrectly numbered as 2-Phenylisopropylamine.

**2-Phenylisovaleric Acid.**

See  $\beta$ : $\beta$ -Dimethylhydrocinnamic Acid.

**Phenylphthalic Acid.**

See Diphenyl-dicarboxylic Acid.

**Phenylpivalic Acid.**

See  $\alpha$ : $\alpha$ -Dimethylhydrocinnamic Acid.

**1-Phenylpropinol-1.**

See Ethinylphenylcarbinol.

**1-Phenylpropionaldehyde.**

See Hydratropic Aldehyde.

**2-Phenylpropyl chloride.**

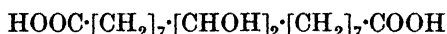
See  $\beta$ -Chlorocumene.

**Phenylpyromellitic Acid.**

See Diphenyl-2:3:5:6-tetracarboxylic Acid.

**Phenylsuccinic Acid.**

*Nitrile*: see  $\beta$ -Cyanohydrocinnamic Acid.

**Phloionic Acid**

$\text{C}_{18}\text{H}_{34}\text{O}_6$  MW, 346

Isolated from cork. Cryst. from MeOH or EtOH. M.p. 124°. Spar. sol.  $\text{Et}_2\text{O}$ , cold  $\text{CHCl}_3$ . Insol. cold  $\text{H}_2\text{O}$ .

*Di-Me ester*:  $\text{C}_{20}\text{H}_{38}\text{O}_6$ . MW, 374. Plates from 70% MeOH. M.p. 77–8°.

*p*:*p*'-Dimethylcarbanilide: needles from EtOH. M.p. 179–80°.

Zetsche et al., *J. prakt. Chem.*, 1938, 150, 140; *Helv. Chim. Acta*, 1931, 14, 632, 846.

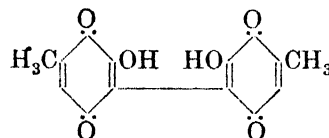
**Phloionolic Acid**

$\text{C}_{18}\text{H}_{36}\text{O}_5$  MW, 332

Isolated from cork. Needles from MeOH, 80% EtOH or AcOEt. M.p. 104°. An enantiomorph, with m.p. 95°, is gradually converted to stable form on standing.

*Me ester*: cryst. from 70% MeOH. M.p. 77°. *p*:*p*'-Dimethylcarbanilide: needles from EtOH. M.p. 155–6°.

Zetsche et al., *J. prakt. Chem.*, 1938, 150, 140; *Helv. Chim. Acta*, 1931, 14, 849.

**Phoenicine** (2:2'-Dihydroxy-4:4'-ditoluquinone)

$\text{C}_{14}\text{H}_{10}\text{O}_6$  MW, 274

Pigment from *Penicillium phoeniceum* and *P. rubrum*. Yellowish brown plates from EtOH, prisms from  $\text{C}_6\text{H}_6$ . M.p. 231–2°.  $\text{FeCl}_3 \rightarrow$  violet col. Yellowish red sols. at  $p_H$  1.8–3.5, reddish violet at  $p_H$  5.4–6.4.

*Diacetyl*: yellow plates from EtOH.Aq. M.p. 117–18°.

*Cyclopentadiene add. comp.*:  $\text{C}_{24}\text{H}_{22}\text{O}_6$ . Colourless needles from EtOH. M.p. 181° decomp.

*Hydroquinone add. comp.*:  $\text{C}_{14}\text{H}_{10}\text{O}_6 \cdot 2\text{C}_6\text{H}_6\text{O}_2$ . Red plates from  $\text{H}_2\text{O}$ . M.p. 198–200°.

*Tetrahydro deriv.*: leucophoenicin. Colourless needles from  $\text{H}_2\text{O}$ . M.p. 247° decomp.

Posternak, *Helv. Chim. Acta*, 1938, 21, 1326.

Friedheim, *ibid.*, 1464.

Curtin, Fitzgerald, Reilly, *Biochem. J.*, 1940, 34, 1605.

**Phthalamic Acid.**

*Nitrile*: see under *o*-Cyanobenzoic Acid.

**Picoline-dicarboxylic Acid.**

See Methylidinicotinic Acid and Methylquinolinic Acid.

**Picolylamine.**

See Aminopicoline.

**Picrasmin**

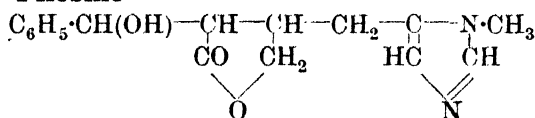
$\text{C}_{22}\text{H}_{30}\text{O}_6$  MW, 390

Bitter constituent of *Picrasma* or *Picraena excelsa*. Colourless rectangular plates and rods from MeOH.Aq. M.p. 218°.  $[\alpha]_D^{20} + 45.4^\circ$  in  $\text{CHCl}_3$ . Contains two methoxy groups.  $\text{AcOH-HCl} \rightarrow$  quassinol,  $\text{C}_{20}\text{H}_{24}\text{O}_6$ , m.p. 263°.  $\text{CrO}_3\text{-AcOH} \rightarrow$  isoquassin,  $\text{C}_{22}\text{H}_{30}\text{O}_6$ , m.p. 221°.

3.5% HCl  $\rightarrow$  semidemethoxyquassin,  $C_{21}H_{28}O_6$ , m.p. 213°.

Clark, *J. Am. Chem. Soc.*, 1938, **60**, 1146.

### Pilosine



Proposed structure

$C_{16}H_{18}O_3N_2$

MW, 286

Alkaloid from *Pilocarpus microphyllus*. Plates from EtOH. M.p. 187°. Sol. hot  $H_2O$ . Very spar. sol.  $Et_2O$ ,  $CHCl_3$ ,  $AcOEt$ ,  $Me_2CO$ ,  $C_6H_6$ .  $[\alpha]_D^{20} + 40.2^\circ$  in  $CHCl_3$ .

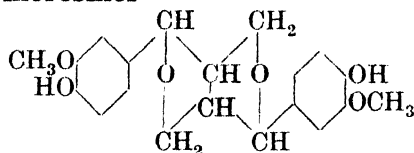
*Sulphate*: plates from EtOH. M.p. 194–5°.  $[\alpha]_D^{20} + 21.0^\circ$  in  $H_2O$ .

*Chloraurate*: golden plates from AcOH. M.p. 143–4°.

Polyakova, Preobrazhenskii, Preobrazhenskii, *J. Gen. Chem. U.S.S.R.*, 1939, **9**, 1402.

Pyman, *J. Chem. Soc.*, 1912, 2260.

### d-Pinoresinol



$C_{20}H_{22}O_6$

MW, 358

Constituent of fir resin. Cryst. from EtOH.Aq. M.p. 122°.  $[\alpha]_D^{21} + 84.4^\circ$  in  $Me_2CO$ .

*Diacetyl*: m.p. 166–167.5°.  $[\alpha]_D^{20} + 50.9^\circ$  in  $Me_2CO$ ,  $+ 49.1^\circ$  in  $CHCl_3$ .

*Dibenzoyl*: m.p. 163–4°.  $[\alpha]_D^{20} + 42.8^\circ$  in  $Me_2CO$ ,  $+ 46.9^\circ$  in  $CHCl_3$ .

*Di-Me ether*:  $C_{22}H_{26}O_6$ . MW, 386. M.p. 107–8°.  $[\alpha]_D^{20} + 64.5^\circ$  in  $CHCl_3$ .

*Di-Et ether*:  $C_{24}H_{30}O_6$ . MW, 414. M.p. 122°.  $[\alpha]_D^{20} + 62^\circ$  in  $CHCl_3$ .

Bamberger, Landsiedl, *Monatsh.*, 1897, **18**, 481.

Kaku, Ri, *J. Pharm. Soc. Japan*, 1937, **57**, 1015.

Erdtmann, *Ann.*, 1935, **516**, 162.

Haworth, *J. Chem. Soc.*, 1942, 448.

### Pinosylvlin.

See 3 : 5-Dihydroxystilbene.

### Piperidine-2 : 6-dicarboxylic Acid.

See Hexahydrodipicolinic Acid.

### 4-[ $\omega$ -4-Piperidylpropyl]-quinoline.

See Rubatoxan.

### Polyporenic Acid A

$C_{30}H_{48}O_4$

MW, 472

Isolated from the fungus, *Polyporus betulinus*,

Fr. Needles from MeOH.Aq.,  $Me_2CO$ .Aq. or dil. AcOH. M.p. 194°. Spar. sol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ .  $[\alpha]_D^{20} + 69^\circ$  in Py. Tetranitromethane in  $CHCl_3 \rightarrow$  yellow col.  $H_2SO_4$ - $CHCl_3 \rightarrow$  red col. Liebermann-Burchard reagent  $\rightarrow$  red col. with green fluor.

*Me ester*:  $C_{31}H_{50}O_4$ . MW, 486. Needles from MeOH. M.p. 142°.  $[\alpha]_D^{20} + 77^\circ$  in  $CHCl_3$ . Contains 2 active H atoms and 2 ethylenic linkages. *Monoformyl*: plates from MeOH. M.p. 148°.  $[\alpha]_D^{20} + 84^\circ$  in  $CHCl_3$ . *Monoacetyl*: needles from MeOH. M.p. 112°.  $[\alpha]_D^{20} + 88^\circ$  in  $CHCl_3$ .

Cross, Eliot, Heilbron, Jones, *J. Chem. Soc.*, 1940, 632.

Cross, Jones, *ibid.*, 1491.

### Polyporenic Acid B

$C_{30}H_{48}O_4$

MW, 472

Constituent of the fungus, *Polyporus betulinus*, Fr. Asbestos-like mass from  $Me_2CO$  or AcOH. M.p. 300–10° decomp. Spar. sol. cold Py. Prac. insol.  $Et_2O$ ,  $C_6H_6$ ,  $CHCl_3$ . Tetranitromethane in  $CHCl_3 \rightarrow$  yellow col.  $H_2SO_4$ - $CHCl_3 \rightarrow$  orange col. Liebermann-Burchard reagent  $\rightarrow$  violet col.

*Me ester*:  $C_{31}H_{50}O_4$ . MW, 486. Needles from MeOH.Aq. Softens at 155°, m.p. 160°. Contains 2 active H atoms and 2 ethylenic linkages.

Cross, Eliot, Heilbron, Jones, *J. Chem. Soc.*, 1940, 632.

### Pomiferin

$C_{25}H_{24}O_6$

MW, 420

Pigment from the fruit of the osage orange tree (*Maclura pomifera*, Raf.). Yellow cryst. from xylene,  $C_6H_6$  or EtOH. M.p. 200–5°.

*Di-Me ether*:  $C_{27}H_{28}O_6$ . MW, 448. Yellow needles from EtOH. M.p. 132°. *Monoacetyl*: needles from EtOH. M.p. 128–9°.

*Tri-Me ether*:  $C_{28}H_{30}O_6$ . MW, 462. Cryst. from EtOH. M.p. 139–5°.

*Diacetyl*: yellow plates from MeOH.Aq. M.p. 134–5°.

*Triacetyl*: needles from EtOH or  $C_6H_6$ -pet. ether. M.p. 154°.

*Di-p-toluenesulphonyl*: cryst. from EtOH. M.p. 148°.

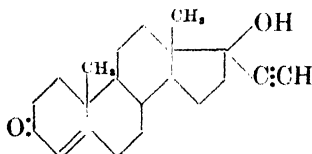
Wolf from, Benton, Gregory, Hess, Mahan, Morgan, *J. Am. Chem. Soc.*, 1939, **61**, 2833.

Wolf from, Gregory, *J. Am. Chem. Soc.*, 1940, **62**, 651.

Wolf from, Mahan, *J. Am. Chem. Soc.*, 1941, **63**, 1253.

**Populnetin**C<sub>14</sub>H<sub>8</sub>O<sub>6</sub> MW, 272Aglucone of populnin. M.p. 270-5°. FeCl<sub>3</sub> → pale green col.*Tetra-acetyl deriv.*: m.p. 127-9°.Neelakantam, Seshadri, *Current Science*, 1938, 7, 16, (*Chem. Abstracts*, 1938, 32, 8486).**Populnin**C<sub>20</sub>H<sub>18</sub>O<sub>11</sub> MW, 434Pigment isolated from Indian tulip flowers. M.p. 228-30° decomp. Hyd. → glucose + populnetin. FeCl<sub>3</sub> → pale green col.

See previous reference.

**Pregneninolone (17-Ethynyltestosterone)**C<sub>21</sub>H<sub>28</sub>O<sub>2</sub> MW, 312Cryst. from CHCl<sub>3</sub>-Me<sub>2</sub>CO or AcOEt. M.p. 270-2° corr. [α]<sub>D</sub><sup>20</sup> + 22.5° in dioxan. Absorption maximum at 2385 Å. More effective than progesterone for oral administration.*Oxime*: cryst. from EtOH.Aq. M.p. 234-5° decomp.*Semicarbazone*: cryst. from Me<sub>2</sub>CO. M.p. 230-1° decomp.Inhoffen, *Ber.*, 1939, 72, 595.Ruzicka, Hofmann, Meldahl, *Helv. Chim. Acta*, 1938, 21, 371.Ruzicka, Hofmann, *Helv. Chim. Acta*, 1937, 20, 1280.**Primetin.**

See 5 : 8-Dihydroxyflavone.

**Prontosil.**

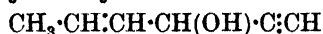
See also Sulphanilamide.

**Propenylacetylene (4-Methylvinylacetylene)**C<sub>5</sub>H<sub>6</sub> MW, 66B.p. 62°. D<sub>4</sub><sup>20</sup> 0.759. n<sub>D</sub><sup>20</sup> 1.438.Lespieau, Journaud, *Bull. soc. chim.*, 1931, 49, 1423.**Propenyl-tert.-butylcarbinol.**

See 5 : 5-Dimethyl-2-hexenol-4.

**Propenylcarbinol.**

See Crotyl Alcohol.

**Propenylethynylcarbinol**C<sub>6</sub>H<sub>8</sub>O MW, 96B.p. 154-6°, 102-3°/111 mm., 75°/24 mm. n<sub>D</sub><sup>25</sup> 1.4651.*Hg comp.*: prisms. M.p. above 360°.*Acetyl*: b.p. 110-12°/100 mm. n<sub>D</sub><sup>20</sup> 1.4463.*Phenylurethane*: needles from pet. ether. M.p. 65°.*β-Naphthylurethane*: needles from pet. ether or MeOH.Aq. M.p. 89°.Jones, McCombie, *J. Chem. Soc.*, 1942, 734.Lespieau, Lombard, *Bull. soc. chim.*, 1935, 2, 369.**2-Propenylpiperidine.**

See β-Coniceine.

**Propionylcatechol.**

See Dihydroxypropiophenone.

**Propionylhydroquinone.**

See Dihydroxypropiophenone.

**2-Propionylpiperidine.**

See Conhydrinone.

**Propionylresorcinol.**

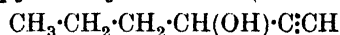
See Dihydroxypropiophenone.

**Propiophenone-β-carboxylic Acid.**

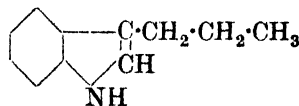
See 1-Benzoylpropionic Acid.

**Propylene bromohydrin.**

See 1-Bromoisopropyl Alcohol.

**n-Propylethynylcarbinol (1-Hexinol-3)**C<sub>6</sub>H<sub>10</sub>O MW, 98

B.p. 55-60°/10 mm.

I.G., E.P. 508,062 (*Chem. Abstracts*, 1940, 34, 447).**3-Propylindole**C<sub>11</sub>H<sub>13</sub>N MW, 159

B.p. 162-4°/20 mm.

*Picrate*: red needles from pet. ether-C<sub>6</sub>H<sub>6</sub>. M.p. 113-14°.Cornforth, Robinson, *J. Chem. Soc.*, 1942, 681.**Propyl naphthyl Ketone.**

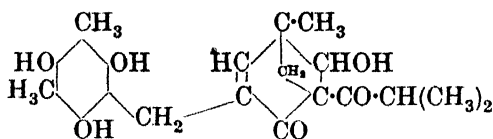
See Butyronaphthone.

**Propylresorcinol.**

See 2 : 4- and 2 : 6-Dihydroxypropylbenzene, and Divarinol.

**1-Propylvaleraldehyde.**

See Di-n-propylacetaldehyde.

**Protokosin**


Probable structure

 $C_{22}H_{28}O_7$ 

MW, 404

Isolated from the dried female flowers of *Hagenia abyssinica* (= *Brayera anthelmintica*). Needles from  $CHCl_3$ -EtOH. M.p. 182°. Sol.  $Et_2O$ ,  $CHCl_3$ ,  $Me_2CO$ , AcOEt. Spar. sol. EtOH, pet. ether. Insol.  $H_2O$ .  $[\alpha]_D + 8.0^\circ$  in  $CHCl_3$ . Possesses no vermifugal properties. Sol. conc.  $H_2SO_4$  with pale green col.  $\rightarrow$  deep red on warming. Alc.  $FeCl_3 \rightarrow$  reddish brown col.

Hems, Todd, *J. Chem. Soc.*, 1937, 562.

**Protostephanine**
 $C_{21}H_{25}O_4N$ 

MW, 355

Alkaloid from *Stephania japonica*, Miers. Prisms +  $1\frac{1}{2}$  MeOH from MeOH. M.p. 75°, 90-5° solvent free. Optically inactive. Contains 1 N- $CH_3$  and 4 O- $CH_3$  groups.

$B, HCl$ : decomp. at 150°.

$B_2, H_2PtCl_6$ : orange prisms. Decomp. at 223°.

*Methiodide*: prisms from  $H_2O$ . M.p. 220-1°.

*Methomethylsulphate*: prisms from  $H_2O$ . Sinters at 227°. M.p. 235°.

Kondo, Watanabe, *J. Pharm. Soc. Japan*, 1938, 58, 46.

**Protoveratridine**
 $C_{31}H_{49}O_9N$ 

MW, 579

Alkaloid from *Veratrum album*. Cryst. M.p. 266-7° corr. Spar. sol. all org. solvents.

$B, HCl$ : cryst. from EtOH. M.p. 243-5° corr. decomp.

*Methylethylglycollic ester*: see Germerine.

*Picrate*: yellow needles from  $Me_2CO$ - $Et_2O$ . Decomp. at 244-6° corr.

*Chloroplatinate*: cryst. from EtOH.Aq. Decomp. at 195-200° corr.

Poethke, *Arch. Pharm.*, 1937, 275, 571.

**Protoveratrine**
 $C_{40}H_{63}O_{14}N$ 

MW, 781

Alkaloid from *Veratrum album*. Cryst. from EtOH. M.p. 255-6° corr. decomp.  $[\alpha]_D^{20} - 9.1^\circ$  in  $CHCl_3$ .

$B, HCl$ : plates. M.p. 234-6° corr. decomp.

$B, HBr$ : prisms. M.p. 230-2° corr. decomp.

$B, HI$ : prisms. M.p. 247-8° corr. decomp.

$B, HSCN$ : prisms. M.p. 221-3° corr. decomp.

$B, HAuCl_4$ : yellow cryst. Decomp. at 199° corr.

*Picrate*: yellow needles. M.p. 216-20° corr. decomp.

Poethke, *Arch. Pharm.*, 1937, 275, 571.

**Pterobilin**
 $C_{33}H_{34}O_6N_4$ 

MW, 582

Wing pigment of butterflies *Pteris brassicae*, *P. rapae*, *P. napi*, *Catopsilia rurina* and *C. statira*. Dark blue rods from AcOEt. Does not melt or decomp. at 310°. Conc.  $HNO_3$  on  $CHCl_3$  sol.  $\rightarrow$  blue col. eventually becoming colourless after passing through green, orange, yellow, etc.

*Di-Me ester*:  $C_{35}H_{38}O_6N_4$ . MW, 610. M.p. 234°. *Zn salt*: green needles. Decomp. above 300°.

Wieland, Tartter, *Ann.*, 1940, 545, 197.

**Punicic Acid**
 $CH_3 \cdot [CH_2]_3 \cdot [CH:CH]_3 \cdot [CH_2]_7 \cdot COOH$ 
 $C_{18}H_{30}O_2$ 

MW, 278

Geometrical isomer of eläostearic acid isolated from oil of pomegranate seeds. Cryst. from pet. ether. M.p. 44°.  $D_4^{20}$  0.9027.  $n_D^{20}$  1.5114. Readily absorbs atmospheric oxygen. Irradiation of xylene sol. for 4 hours  $\rightarrow$   $\beta$ -eläostearic acid.  $H(+Pt) \rightarrow$  stearic acid.  $KMnO_4 \rightarrow$  azelaic, oxalic and valeric acids.

Toyama, Tsuchiya, *Chem. Abstracts*, 1935, 29, 5294.

Farmer, van den Heuvel, *J. Chem. Soc.*, 1936, 1809.

Toyama, Uozaki, *Chem. Abstracts*, 1937, 31, 249.

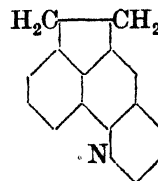
**Purapurine.**

See Solasonine.

**Pyridoxin.**

See Adermin.

4 : 5-[ $\alpha$  :  $\beta'$ -Pyridino]-acenaphthene (*Acenaphthapyridine*, *acenaphthaquinoline*)


 $C_{15}H_{11}N$ 

MW, 205

Needles from  $Et_2O$ . M.p. 67°. Sol. EtOH,  $Et_2O$ , AcOH,  $CHCl_3$ ,  $C_6H_6$ . Sol. dil. AcOH



with blue fluor. Sol. conc.  $\text{H}_2\text{SO}_4$  with yellowish green col.

$\text{B.HCl}$ : yellow needles from EtOH. M.p. 305°.

$\text{B.H}_2\text{SO}_4$ : yellow needles from EtOH. M.p. 238°.

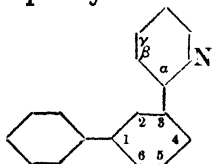
*Methiodide*: yellow needles from MeOH. M.p. above 315°.

*Picrate*: m.p. 234°.

Zinke, Raith, *Monatsh.*, 1919, 40, 273.

Stewart, *J. Chem. Soc.*, 1925, 127, 1331.

### 3- $\alpha$ -Pyridyldiphenyl



$\text{C}_{17}\text{H}_{13}\text{N}$

MW, 231

Viscous oil. B.p. 75–85°/0.002 mm. Absorption maximum: 2480 Å. in hexane.

*Picrate*: yellow prisms from  $\text{Me}_2\text{CO}$ . M.p. 169°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1281.

Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

### 3- $\beta$ -Pyridyldiphenyl

Viscous oil. B.p. 75–85°/0.002 mm. Absorption maximum: 2455 Å. in hexane.

*Picrate*: needles from  $\text{Me}_2\text{CO}$ . M.p. 178–9°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1281.

Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

### 3- $\gamma$ -Pyridyldiphenyl

Plates from pet. ether. M.p. 81–2°. Absorption maximum: 2480 Å. in hexane.

*Picrate*: needles from  $\text{Me}_2\text{CO}$ . M.p. 231°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1282.

Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

### 4- $\alpha$ -Pyridyldiphenyl

Plates from EtOH. M.p. 141–2°. Absorption maximum: 2920 Å. in hexane.

*Picrate*: needles from  $\text{Me}_2\text{CO}$ . M.p. 186–7°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1282.

Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

### 4- $\beta$ -Pyridyldiphenyl

Plates from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 151–2°. Absorption maximum: 2790 Å. in hexane.

*Picrate*: needles from  $\text{Me}_2\text{CO}$ . M.p. 208–10°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1282.

Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

### 4- $\gamma$ -Pyridyldiphenyl

Plates from  $\text{C}_6\text{H}_6$ -pet. ether. M.p. 209°. Absorption maximum: 2790 Å. in hexane.

*Picrate*: needles from  $\text{Me}_2\text{CO}$ . M.p. 215°.

Heilbron, Hey, Lambert, *J. Chem. Soc.*, 1940, 1283.

Gillam, Hey, Lambert, *J. Chem. Soc.*, 1941, 364.

### Pyridylmethylamine.

See Aminopicoline.

### Pyridylsulphanilamide.

See Sulphapyridine.

### Pyrimidone-imide.

See Aminopyrimidine.

### Pyrimidylsulphanilamide.

See Sulphadiazine.

### Pyrocinchonic Acid.

See Dimethylmaleic Acid.

## Q

### Quassin

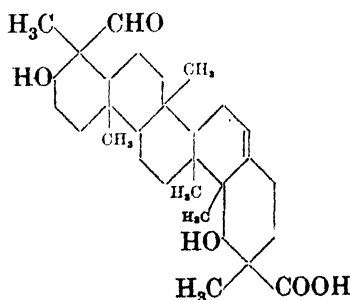
$\text{C}_{22}\text{H}_{30}\text{O}_6$

MW, 390

Bitter constituent of quassia or Surinam wood (*Quassia amara*). Rods and plates from MeOH.Aq. M.p. 205–6°.  $[\alpha]_D^{20} + 39.8^\circ$  in  $\text{CHCl}_3$ . Contains one active H atom and two  $\text{CH}_3\text{O}$  groups. Conc. HBr or HCl  $\longrightarrow$  quassinol,  $\text{C}_{20}\text{H}_{24}\text{O}_6$ , m.p. 263°.  $\text{CrO}_3 \longrightarrow$  isoquassin,  $\text{C}_{22}\text{H}_{30}\text{O}_6$ , m.p. 221°.

Clark, *J. Am. Chem. Soc.*, 1937, 59, 927, 2511; 1938, 60, 1146.

### Quillaic Acid



Suggested structure

$\text{C}_{30}\text{H}_{46}\text{O}_5$

MW, 48

Sapogenin obtained on hydrolysis of quillaia saponin. Needles from EtOH. M.p. 294°.  $[\alpha]_D$

+ 56.1° in Py. Tetranitromethane → yellow col.

*Me ester*:  $C_{31}H_{48}O_5$ . MW, 500. Needles from MeOH. M.p. 225°.  $[\alpha]_D + 40.5^\circ$  in Py.

*Oxime*: needles. M.p. 238°.

*Diacetyl*: needles from AcOH. M.p. 250°.

*Oxime*: needles from MeOH. M.p. 282°.

*Semicarbazone*: needles. M.p. 288°.

Windaus, Hampe, Rabe, *Z. physiol. Chem.*, 1926, 160, 301.

Elliott, Kon, *J. Chem. Soc.*, 1939, 1130.

Bilham, Kon, *J. Chem. Soc.*, 1940, 1471; 1941, 552.

Bilham, Kon, Ross, *J. Chem. Soc.*, 1942, 532.

### Quinaldine-sulphonic Acid.

See Methylquinoline-sulphonic Acid.

### Quinaldylamine.

See Aminomethylquinoline.

### Quinethyline.

See under Cupreine.

### Quinisoamyline.

See under Cupreine.

### Quinisopropylene.

See under Cupreine.

**Quinizarin-5-carboxylic Acid** (5 : 8-Di-hydroxyanthraquinone-1-carboxylic acid, 1 : 4-dihydroxyanthraquinone-5-carboxylic acid).

Red needles from *p*-xylene. Decomp. at 250°. Sol. EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Spar. sol. H<sub>2</sub>O.

Kögl, Deijis, *Ann.*, 1935, 515, 31.

**Quinizarin-6-carboxylic Acid** (5 : 8-Di-hydroxyanthraquinone-2-carboxylic acid, 1 : 4-dihydroxyanthraquinone-6-carboxylic acid).

Orange brown needles from AcOH. Sol. Na<sub>2</sub>CO<sub>3</sub> → violet red col. Sol. NaOH → blue col.

Bayer, D.R.P. 84,505.

### Quinpropylene.

See under Cupreine.

### *p*-Quinquiphenyl.

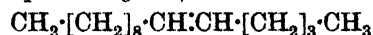
See *p*-Pentaphenyl.

## R

### 2-R Acid.

See 2-Amino-8-naphthol-3 : 6-disulphonic Acid.

**Renghol** (*Dihydrourushiol*, 15-[2 : 3-dihydroxy-phenyl]-5-pentadecylene)



$C_{21}H_{34}O_2$

MW, 318

Dict. of Org. Comp.—111.

Toxic constituent of renghas fruit (*Semecarpus heterophylla*, Bl.). M.p. 14–15°. B.p. 170–2°/0.0001 mm. Ozonolysis → valeraldehyde. Vesicant.

*Me ether*:  $C_{22}H_{36}O_2$ . MW, 332. B.p. 160°/0.0001 mm.

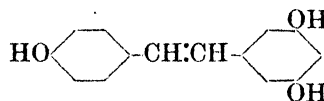
*Di-Me ether*:  $C_{23}H_{38}O_2$ . MW, 346. B.p. 226°/5 mm., 155–7°/0.0001 mm.

Backer, Haack, *Rec. trav. chim.*, 1938, 57, 225.

### Resorcitol.

See Hexahydroresorcinol.

### Resveratrol (3 : 5 : 4'-Trihydroxystilbene)



$C_{14}H_{12}O_3$

MW, 228

Obtained from roots of white hellebore (*Veratrum grandiflorum*, Loes. fil.). Cryst. from EtOH.Aq. M.p. 261° decomp. Absorption maxima at 3610, 3350, 3020, 2900 and 2480 Å. FeCl<sub>3</sub> → dark green col.

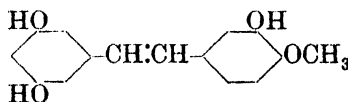
*Triacetyl*: needles from EtOH. M.p. 114–16°.

*Tri-Me ether*: cryst. from MeOH. M.p. 56–7°. B.p. 140–60°/0.01 mm.

Takaoka, *Chem. Abstracts*, 1940, 34, 7887; 1941, 35, 1398.

Späth, Kromp, *Ber.*, 1941, 74, 867.

**Rhapontigenin** (3 : 4 : 3' : 5'-Tetrahydroxy stilbene 4-methyl ether)



$C_{15}H_{14}O_4$

MW, 258

Aglucone from rhapontin from rhapontic spice. M.p. 186–7°.

*Triacetyl*: (1) M.p. 114°. (2) M.p. 128°.

*Tribenzoyl*: m.p. 142°.

Kawamura, *J. Pharm. Soc. Japan*, 1938, 58, 405.

Takaoka, *Proc. Imper. Acad., Tokyo*, 1940, 16, 408.

### Rhapontin

$C_{21}H_{24}O_9$

MW, 420

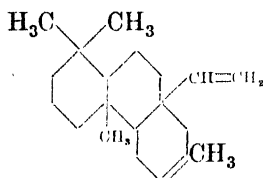
Glycoside from rhapontic spice. Decomp. at 236–7°.  $[\alpha]_D^{25} - 59.5^\circ$  in Me<sub>2</sub>CO. Hyd. → glucose + rhapontigenin.

*Acetyl deriv.*: m.p. 135–6°.

Kawamura, *J. Pharm. Soc. Japan*, 1938, 58, 405.

**Riboflavine.**

See Lactoflavine.

**Rimuene**

Suggested formula

 $C_{20}H_{32}$ 

MW, 272

From leaves of *Dacrydium cupressinum* (Salander), etc. Cryst. from EtOH. M.p. 55°.  $[\alpha]_D^{25} + 44.7^\circ$  in  $CHCl_3$ . Se dehydrogenation  $\rightarrow$  pimanthrene.

**Hydrochloride**:  $C_{20}H_{33}Cl$ . M.p. 63°.

**Nitroso-chloride**:  $C_{20}H_{32}ONCl$ . M.p. 86–8°.

**Tetrabromide**: m.p. 55–60°.

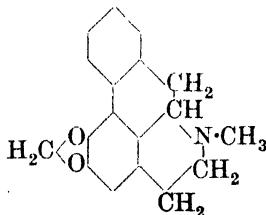
McDowall, Finlay, *J. Soc. Chem. Ind.*, 1925, **44**, 42r.

Beath, *J. Soc. Chem. Ind.*, 1933, **52**, 338r.

Brandt, *Chem. Abstracts*, 1939, **33**, 551.

**Rivanol.**

See under 7-Hydroxy-2:5-diaminoacridine.

**Roemerine** $C_{18}H_{17}O_2N$ 

MW, 279

Alkaloid from *Roemaria refracta*, D.C. M.p. 102–3°.  $[\alpha]_D^{25} - 77.18^\circ$  in EtOH.

**B.HCl**: m.p. 262–3°.

**Methiodide**: m.p. 215–16°.

**Picrate**: m.p. 195–6°.

Konovalova, Junusov, Orékhov, *J. Gen. Chem. U.S.S.R.*, 1939, **9**, 1868; *Bull. soc. chim.*, 1939, **6**, 1479; 1940, **7**, 70.

**Rottlerin** $*C_{30}H_{28}O_8$ 

MW, 516

Isolated from "kamala," an Indian colouring matter and anthelmintic drug, also from *Rottlera tinctoria*, Roxb. Yellowish brown cryst. from toluene or AcOEt. M.p. 212° (201–2°).  $O_3$  or heating with dil. alkali  $\rightarrow$   $C_6H_5 \cdot CHO$ .  $H_2O_2$   $\rightarrow$  cinnamic acid. Heat with EtOH or AcOH  $\rightarrow$  isorottlerin, m.p. 180°.

**Di-Me ether**: pale yellow leaflets from  $CHCl_3$ -MeOH or AcOEt-Me<sub>2</sub>CO. M.p. 245–6° decomp.

**Penta-Me ether**: cryst. from pet. ether. M.p. 144°.

**Penta-acetyl deriv.**: plates from  $C_6H_6$ -EtOH or Me<sub>2</sub>CO-EtOH. M.p. 214°.

Dutt, *J. Chem. Soc.*, 1925, **127**, 2044.

Brockmann, Maier, *Ann.*, 1938, **535**, 149.

Rojahn, *Chem. Abstracts*, 1937, **31**, 6816.

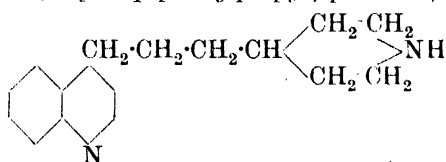
**Rotundifoline** $C_{22}H_{26}O_5N_2$ 

MW, 398

Alkaloid from *Mitragyna rotundifolia*. Prisms from MeOH. M.p. 233–4°. Sol.  $CHCl_3$ . Mod. sol. Me<sub>2</sub>CO, EtOH,  $C_6H_6$ . Spar. sol. Et<sub>2</sub>O, AcOEt.  $[\alpha]_D^{25} + 124^\circ$  in  $CHCl_3$ .  $FeCl_3 \rightarrow$  red col. Hyd.  $\rightarrow$  rotundifolic acid, decomp. about 165°.

Barger, Dyer, Sargent, *J. Org. Chem.*, 1939, **4**, 418.

**Rubatoxan** (1-[4-Piperidyl]-3-[4-quinolyl]-propane, 4-[ω-4-piperidylpropyl]-quinoline)

 $C_{17}H_{22}N_2$ 

MW, 254

Yellowish liq. B.p. 185°/0.02 mm.

**B,2HCl**: cryst. from EtOH-AcOEt. M.p. 197°.

**Chloroplatinate**: orange cryst. from EtOH. M.p. above 360°.

**Dipicrate**: cryst. from EtOH. M.p. 203–5°.

Prelog, Seiwerth, Hahn, Cerkovnikov, *Ber.*, 1939, **72**, 1325.

**Rubijervine** $C_{26}H_{43}O_2N$ 

MW, 401

Alkaloid from *Veratrum album*. Cryst. +  $H_2O$  from EtOH or Me<sub>2</sub>CO. M.p. 239–40° decomp. Conc.  $H_2SO_4 \rightarrow$  green col.

**B,HI**: needles from Me<sub>2</sub>CO-Et<sub>2</sub>O. M.p. 261–2° decomp.

**p-Bromobenzoyl**: cryst. from EtOH- $C_6H_6$ . M.p. 254–6° decomp.

Poethke, *Arch. Pharm.*, 1938, **276**, 170.

**Rubradinine (Mitraphyline)** $C_{24}H_{28}O_4N_2$ 

MW, 408

Alkaloid from *Adina rubrostipulata*, K. Schum. Needles from 95% EtOH. M.p. 306°.  $[\alpha]_D^{25} - 22.3^\circ$  in  $CHCl_3$ .

**B,H<sub>2</sub>SO<sub>4</sub>,5H<sub>2</sub>O**: m.p. 245°.

**Picrate**: m.p. 166°.

Denis, *Chem. Abstracts*, 1937, **31**, 3928; *Brit. Chem. Abstracts*, 1940, A II, 262.

**Rubrofusarin**

$C_{15}H_{12}O_5$  MW, 272

Pigment of *Fusarium culmorum* Sacc., and related forms. Orange red needles from pet. ether,  $C_6H_6$  or EtOH. M.p. 210–11°. Insol.  $H_2O$ . Spar. sol. aq. alkalis.  $FeCl_3 \rightarrow$  greenish brown col.

*Monoacetyl deriv.*: golden yellow hexagonal prisms from  $C_6H_6$  or AcOH. M.p. 211°.

*Diacetyl deriv.*: prac. colourless rods. M.p. 260°.

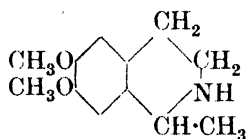
*Me ether*: m.p. 203–4°.

*Di-Me ether*: m.p. 187–8°. *Ferrichloride*: maroon prisms from AcOH. M.p. 183–4° decomp.

Ashley, Hobbs, Raistrick, *Biochem. J.*, 1937, 31, 385.

**S**

**Salsolidine** (O-Methylsalsoline, 6:7-dimethoxy-1-methyl-1:2:3:4-tetrahydroisoquinoline)



$C_{12}H_{17}O_2N$  MW, 207

l. Alkaloid from *Salsola richteri*. Plates from  $H_2O$ . M.p. 47.5–48.5° (60–2°, anhyd. 71–3°). B.p. about 120°/0.01 mm.  $[\alpha]_D^{25} - 59.7^\circ$  in EtOH.

*B,HCl*: plates from  $H_2O$ . M.p. 235–6°.  $[\alpha]_D^{25} - 24.8^\circ$  in  $H_2O$ .

*N-Benzoyl*: needles from  $Me_2CO$ . M.p. 124–5°.

*Picrate*: yellow cryst. from EtOH. M.p. 194–5°.

*Picrolonate*: cryst. from EtOH. M.p. 235.5–236° (220–1°).

dl.

M.p. 47.5–48.5°.  $[\alpha]_D^{25} + 59.9^\circ$  in EtOH.

*B,HCl*: m.p. 235–6°.  $[\alpha]_D^{25} + 25.3^\circ$  in  $H_2O$ .

*Picrate*: m.p. 193–4°.

*Picrolonate*: m.p. 235.5–236°.

dl.

M.p. 53–53.5°. B.p. about 140°/1 mm.

*B,HCl*: m.p. 196–7°.

*N-Benzoyl*: cryst. from EtOH. Aq. M.p. 127–8°.

*Picrate*: cryst. from MeOH. M.p. 201–201.5° decomp.

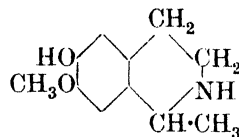
*Picrolonate*: cryst. from MeOH. M.p. 241° decomp.

Det. of Org. Comp.—III.

Späth, Dengel, *Ber.*, 1938, 71, 114.

Proskurnina, Orekhov, *Bull. soc. chim.*, 1937, 4, 1265.

**Salsoline** (6-Hydroxy-7-methoxy-1-methyl-1:2:3:4-tetrahydroisoquinoline)



$C_{11}H_{15}O_2N$  MW, 193

Alkaloid from *Salsola richteri*. Micro-cryst. powder. M.p. 218–21°. Sol. EtOH,  $CHCl_3$ . Spar. sol.  $H_2O$ ,  $C_6H_6$ . Insol. pet. ether. Sol. aq. alkalis.

*B,HCl*: needles +  $1\frac{1}{2}H_2O$ . M.p. 141–52°.

*Benzoyl deriv.*: cryst. from EtOH. M.p. 172–4°.

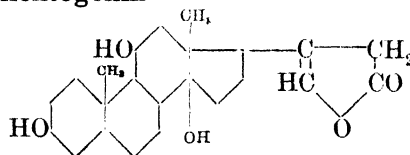
*Dibenzoyl*: cryst. from EtOH. M.p. 166–8°.

*Me ether*: see Salsolidine.

Orekhov, Proskurnina, *Ber.*, 1933, 66, 841; 1934, 67, 878.

Späth, Orekhov, Kuffner, *Ber.*, 1934, 67, 1214.

**Sarmentogenin**



Suggested structure

$C_{23}H_{34}O_5$  MW, 390

Isolated from seeds of *Strophanthus sarmentosus*. Needles from EtOH. M.p. 270°.  $[\alpha]_D^{25} + 21.3^\circ$  in EtOH.

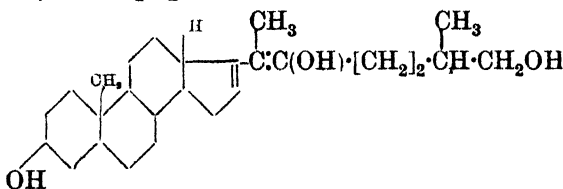
*Dibenzoyl deriv.*: hexagonal prisms from  $Me_2CO$ . Aq. M.p. 281°.  $[\alpha]_D^{25} + 14^\circ$  in  $Me_2CO$ .

Jacobs, Heidelberger, *J. Biol. Chem.*, 1929, 81, 765.

Mason, Hoehn, *J. Am. Chem. Soc.*, 1938, 60, 2824.

Tschesche, Bohle, *Ber.*, 1936, 69, 2497.

**ψ-Sarsapogenin**



Suggested structure

$C_{27}H_{44}O_3$

MW, 416

Needles from AcOEt. M.p. 171-3°. Sol. Et<sub>2</sub>O.

Di-p-nitrobenzoyl: pale yellow cryst. from Me<sub>2</sub>CO. M.p. 156.5-159°.

Marker, Rohrmann, *J. Am. Chem. Soc.*, 1940, **62**, 518, 521.

Marker, Jones, Krueger, *ibid.*, 2532.

### Satisterol

C<sub>27</sub>H<sub>46</sub>O MW, 386

Phytosterol from rice embryo. M.p. 156°.  $[\alpha]_D^{25} = -14.5^\circ$ .

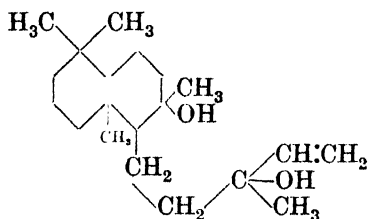
Acetyl: m.p. 111°.  $[\alpha]_D^{25} = -9.7^\circ$ .

Propionyl: m.p. 106°.  $[\alpha]_D^{25} = -6.8^\circ$ .

Benzoyl: m.p. 129°.  $[\alpha]_D^{25} = -14.5^\circ$ .

Kimm, *Sci. Papers Inst. Phys. Chem. Research, Tokyo*, 1938, **34**, 637.

### Sclareol



C<sub>20</sub>H<sub>36</sub>O<sub>2</sub> MW, 308

Diterpene diol from leaves of *Salvia sclarea*, L. Isolated from pet. ether. M.p. 105.5-106° corr. B.p. 182°/1 mm., 163-5°/0.25 mm.  $[\alpha]_D^{25} = -6.25^\circ$  in EtOH.

Ruzicka, Engel, Fischer, *Helv. Chim. Acta*, 1938, **21**, 364.

Ruzicka, Janot, *Helv. Chim. Acta*, 1931, **14**, 645.

Hosking, Brandt, *Ber.*, 1935, **68**, 37.

Janot, *Ann. chim.*, 1932, **17**, 5.

Ruzicka, Seidel, Engel, *Helv. Chim. Acta*, 1942, **25**, 621.

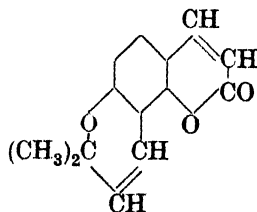
### Senecioaldehyde.

See 2: 2-Dimethylacrolein.

### p-Septiphenyl.

See p-Heptaphenyl.

### Seselin



C<sub>14</sub>H<sub>12</sub>O<sub>3</sub>

MW, 228

Constituent of fruit of *Seles indicum* (Wall.) W. and A. Cryst. from MeOH-Et<sub>2</sub>O. M.p. 119-20°. AcOH-H<sub>2</sub>SO<sub>4</sub> → umbelliferone.

Bose, Guha, *Chem. Zentr.*, 1937, **II**, 238.

Späth, Bose, Matzke, Guha, *Ber.*, 1939, **72**, 821.

Späth, Hillel, *ibid.*, 963, 2093.

### Sesquigoyol

C<sub>15</sub>H<sub>26</sub>O MW, 222

Sesquiterpene alcohol from oil of *Pinus formosana*, Hayata. M.p. 137-137.5°. B.p. 285-9°, 160-5°/8 mm.  $[\alpha]_D^{25} = +93.4^\circ$ .

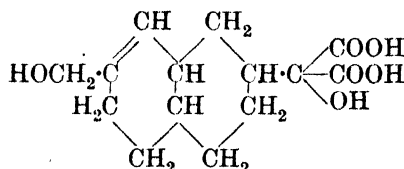
Acetyl: b.p. 152-5°/4 mm.  $n_D^{25} 1.4902$ .  $[\alpha]_D^{25} +22.2^\circ$ .

Yeigai, Sabe, *J. Chem. Soc. Japan*, 1935, **56**, 1118.

### p-Sexiphenyl.

See p-Hexaphenyl.

### Shellolic Acid



C<sub>15</sub>H<sub>20</sub>O<sub>6</sub> MW, 296

Isolated from shellac. Plates from H<sub>2</sub>O. M.p. 206°. Sol. EtOH. Spar. sol. Et<sub>2</sub>O, AcOEt, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>. Insol. petrol.

Di-Me ester: C<sub>17</sub>H<sub>24</sub>O<sub>6</sub>. MW, 224. Prisms from EtOH. M.p. 156°.  $[\alpha]_D^{25} +36^\circ$ .

Di-hydrazide: prisms from H<sub>2</sub>O. M.p. 246° decomp.

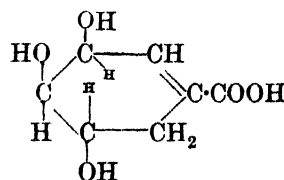
Di-phenylurethane: cryst. from CCl<sub>4</sub>. M.p. about 92-4°.

Kirk, Spoerri, Gardner, *J. Am. Chem. Soc.*, 1941, **63**, 1243.

Nagel, Martens, *Ber.*, 1937, **70**, 2173.

Harries, Nagel, *Ber.*, 1922, **55**, 3833.

### Shikimic Acid



C<sub>7</sub>H<sub>10</sub>O<sub>5</sub>

MW, 174

Constituent of fruits of *Illicium religiosum*, Siebold. Cryst. from H<sub>2</sub>O. M.p. 178-80°. Insol. EtOH, Et<sub>2</sub>O, CHCl<sub>3</sub>.

A,CH<sub>3</sub>NH<sub>2</sub>: m.p. 163-4°.

A,NH<sub>2</sub>·NH<sub>2</sub>: m.p. 147-8°.

$A, C_6H_5NH_2$ : m.p. 194–5°.

$A, Py$ : micro-plates. M.p. 184–5°.

$A, Quinine$ : needles from EtOH. M.p. 221–2°.

$A, Strychnine$ : plates. Sinters at 154°. M.p. 234–6°.

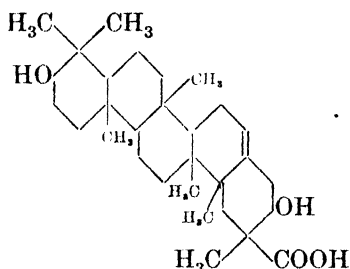
*Me ester*: needles from AcOH–petrol. M.p. 113–14°.

*Triacetyl*: b.p. 200–10°/1 mm.

$Me_2CO$  comp.: cryst. from AcOH–petrol. M.p. 184°.

Fischer, Dangschat, *Naturwissenschaften*, 1938, 26, 562; *Helv. Chim. Acta*, 1937, 20, 705; 1935, 18, 1206; 1934, 17, 1200.

### Siaresinolic Acid



Suggested structure

$C_{30}H_{48}O_4$  MW, 472

Isolated from Siam benzoin (Siamese gum). Plates from MeOH–Et<sub>2</sub>O. M.p. 274–5°.  $[\alpha]_D^{25} + 98.5^\circ$  in CHCl<sub>3</sub>.

*Me ester*: prismatic plates from petrol. M.p. 180–1°.  $[\alpha]_D^{25} + 48.4^\circ$  in CHCl<sub>3</sub>. *Acetyl*: needles from MeOH. M.p. 110–20°.

*Et ester*: needles or plates from petrol. M.p. 108°.

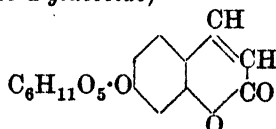
*Bromolactone*: m.p. 178–80°.  $[\alpha]_D^{25} + 151.8^\circ$  in CHCl<sub>3</sub>.

Winterstein, Egli, *Z. physiol. Chem.*, 1931, 202, 207.

Zinke, Lieb, *Monatsh.*, 1918, 39, 95, 627.

Bilham, Kon, Ross, *J. Chem. Soc.*, 1942, 540.

**Skimmin** (7-Hydroxycoumarin d-glucoside, umbelliferone d-glucoside)



$C_{15}H_{16}O_8 \cdot H_2O$  MW, 342

Isolated from wood of *Skimmia Japonica*. M.p. 219–21°.  $[\alpha]_D^{25} - 79.8^\circ$  in Py. 4% H<sub>2</sub>SO<sub>4</sub> → umbelliferone + glucose.

*Tetra-acetyl*: cryst. from MeOH. M.p. 183–4°.  $[\alpha]_D^{25} - 63.3^\circ$  in Py.

Späth, Neufeld, *Rec. trav. chim.*, 1938, 57, 535.

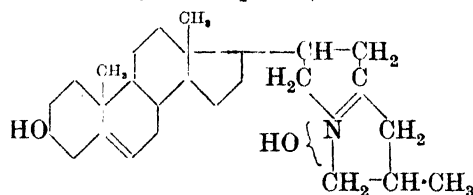
### Solancarpidine.

See Solasodine.

### Solanine-S.

See Solasonine.

### Solasodine (Solancarpidine)



Suggested structure

$C_{27}H_{43}O_2N$  MW, 413

Occurs as glucoside in *Solanum xanthocarpum* and *S. aviculare*. M.p. 197–8°.  $[\alpha]_D^{20} - 92.4^\circ$  in C<sub>6</sub>H<sub>6</sub>. Gives usual sterol colour reactions.

*Monoacetyl*: plates from EtOH–AcOEt. M.p. 195°.

*Monobenzoyl*: m.p. 216–17°.

3:5-Dinitrobenzoyl: yellowish needles from EtOH. M.p. 191.5–193°.

*Hydriodide*: prisms from MeOH–Et<sub>2</sub>O. M.p. 286° decomp.

Rochelmeyer, Chen, *Arch. Pharm.*, 1939, 277, 329.

Briggs, *J. Chem. Soc.*, 1942, 3.

### Solasonine (Solanine-S, purapurine)

$C_{45}H_{73}O_{16}N$  MW, 883

Glycoside isolated from *Solanum aviculare*. Sol. hot alcohols → gels on cooling.  $[\alpha]_D$  about  $-87^\circ$  in EtOH. Hyd. → solasodine, glucose, galactose and rhamnose. Conc. H<sub>2</sub>SO<sub>4</sub> finally → crimson col. with brown fluor.

Briggs, *Nature*, 1939, 144, 247; *J. Chem. Soc.*, 1942, 3.

Levi, *J. Soc. Chem. Ind.*, 1930, 49, 395T.

### Sophoraflavanoloside

$C_{27}H_{30}O_{16}$  MW, 610

Glycoside from green fruits of *Sophora japonica*. Yellow needles. M.p. 207–8°.  $[\alpha]_D^{20} - 61^\circ$  in EtOH. Hyd. → kempferol + sophorose, m.p. 195–6°. Emulsin → d-glucose.

Rabaté, Dussy, *Bull. soc. chim. biol.*, 1938, 20, 459.

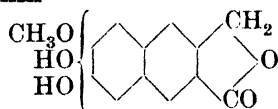
### Sophoricoside (Genistein β-glucoside)

$C_{21}H_{20}O_{10}$  MW, 432

Isolated from green pods of *Sophora japonica*. Prisms from EtOH. M.p. 297°.  $[\alpha]_D^{20} - 46.7^\circ$ . Acid hyd.  $\rightarrow$  genistein + glucose.

*Hexa-acetyl*: cryst. from AcOH or EtOH. M.p. 230°.

Charaux, Rabaté, *Bull. soc. chim. biol.*, 1938, 20, 454; *J. pharm. chim.*, 1935, 21, 546.

 **$\alpha$ -Sorigenin**

Proposed structure

$C_{13}H_{10}O_5$  MW, 246

Pale yellow needles. M.p. 227–9° decomp. Monobasic.

*Di-Me ether*: m.p. 183.5–184.5°.

*Diacetyl*: m.p. 255–9°.

Nikuni, *Chem. Abstracts*, 1940, 34, 3259; 1939, 33, 6301; 1938, 32, 7469.

 **$\alpha$ -Sorinin**

$C_{24}H_{28}O_{14}$  MW, 540

Isolated from bark of *Rhamnus japonica*, Max. Needles from MeOH.Aq. M.p. 159°. Boiling  $H_2O \rightarrow \alpha$ -sorigenin + primeverose.

Nikuni, *Chem. Abstracts*, 1939, 33, 6301; 1938, 32, 7469.

**Soya-sapogenol A**

$C_{30}H_{50}O_4$  MW, 474

Triterpene alcohol from soya bean saponin. Leaflets from MeOH. M.p. 308–12°.  $[\alpha]_D^{25} + 102.3^\circ$  in  $CHCl_3$ .

*Tetra-acetyl*: m.p. 232°.  $[\alpha]_D^{25} + 86.1^\circ$  in  $CHCl_3$ .

*Tribenzoyl*: m.p. 255°.

Tsuda, Kitewaga, *Ber.*, 1938, 71, 790; 1937, 70, 2083.

Miyasaka, *J. Pharm. Soc. Japan*, 1937, 57, 464.

**Soya-sapogenol B**

$C_{30}H_{50}O_3$  MW, 458

Triterpene alcohol from soya bean saponin. Needles from MeOH. M.p. 258–9°.  $[\alpha]_D^{20} + 92.4^\circ$  in  $CHCl_3$ .

*Triformyl*: prisms from  $Me_2CO-Et_2O$ . M.p. 218°.

*Triacetyl*: plates from MeOH. M.p. 175–6°.  $[\alpha]_D^{25} + 83^\circ$  in  $CHCl_3$ . *Dibromide*: needles from  $Me_2CO-MeOH$ . M.p. 225–7°.

*Tribenzoyl*: m.p. 133°.

*Tri-p-bromobenzoyl*: cryst. from  $Me_2CO$ . M.p. 255–7°.

Tsuda, Kitewaga, *Ber.*, 1938, 71, 790; 1937, 70, 2083.

Miyasaka, *J. Pharm. Soc. Japan*, 1937, 57, 464.

**Soya-sapogenol C**

$C_{30}H_{50}O_2$  MW, 442

Triterpene alcohol from soya bean saponin. Needles from MeOH. M.p. 238–9°.  $[\alpha]_D^{21} + 70.7^\circ$  in  $CHCl_3$ .

*Diformyl*: prisms from  $Me_2CO-Et_2O$ . M.p. 265°.

*Diacetyl*: plates from MeOH–EtOH. M.p.

198°. *Dibromide*: needles from  $Me_2CO-Et_2O$ . M.p. 225–7°.

*Dibenzoyl*: cryst. from  $Me_2CO-MeOH$ . M.p. 188°.

Tsuda, Kitewaga, *Ber.*, 1938, 71, 790; 1937, 70, 2083.

Miyasaka, *J. Pharm. Soc. Japan*, 1937, 57, 464.

**Soya-sapogenol D**

$C_{30}H_{50}O_3$  MW, 458

Triterpene alcohol from soya bean saponin. Prisms from  $Me_2CO-MeOH$ . M.p. 298–9°.  $[\alpha]_D^{21} - 60.8^\circ$  in  $CHCl_3$ .

*Diformyl*: needles from  $Me_2CO-Et_2O$ . M.p. 231°.

*Diacetyl*: prisms from MeOH. M.p. 192°.

*Dibenzoyl*: needles from  $Me_2CO$ . M.p. 240°.

Tsuda, Kitewaga, *Ber.*, 1938, 71, 790; 1937, 70, 2083.

Miyasaka, *J. Pharm. Soc. Japan*, 1937, 57, 464.

**Spartioidine**

$C_{18}H_{23}O_5N$  MW, 333

Alkaloid from *Senecio spartioides*. Prisms from MeOH. M.p. 178°.

*Methiodide*: plates from  $CHCl_3$ . M.p. 239° decomp.

Manske, *Can. J. Research.*, 1939, 17, 1.

**Spathulatine**

$C_{33}H_{44}O_5N_4$  MW, 596

Alkaloid from *Lupinus sericeus* and *L. spathulatus*. Cryst. from  $Me_2CO$ . M.p. 233–4° corr. Sol.  $H_2O$ , EtOH,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol.  $Et_2O$ , pet. ether. Sols. are slightly laevorotatory. Sensitive to strong min. acids.

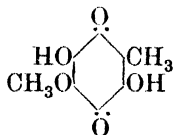
*B,3KI*: prisms from  $H_2O$ . M.p. 260–1°.

*Picrate*: yellow needles. M.p. 182–4°.

**Methiodide** : cryst. M.p. 250-2°.

Couch, *J. Am. Chem. Soc.*, 1940, **62**, 554;  
1924, **46**, 2507.

**Spinulosin** (3 : 6-Dihydroxy-5-methoxytolu-quinone)



$C_8H_8O_5$

MW, 184

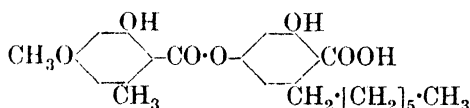
Metabolic product of *Penicillium spinulosum*,  
Tham. Sublimes at 120°/1 mm. in black metallic  
plates, m.p. 202-3°.

**Di-Me ether** :  $C_{10}H_{12}O_5$ . MW, 212. Reddish  
orange needles from EtOH. M.p. 80°.

**Diacetyl** : needles from EtOH. M.p. 139-5°.

Anslow, Raistrick, *Biochem. J.*, 1938, **32**,  
803; *J. Chem. Soc.*, 1939, 1446.

### Sphaerophorin



$C_{23}H_{27}O_7$

MW, 415

Depside from the lichen *Sphaerophorus melano-*  
*carpus*. Plates or needles from  $C_6H_6$ . M.p.  
140°. Alc. KOH  $\rightarrow$  everninic acid + 5-heptyl-  
resorcinol, m.p. 57-8°.

**Diacetyl** : needles from 80% EtOH. M.p.  
133-4°.

**Di-Me ether-Me ester** : prisms from 80%  
EtOH. M.p. 85-6°.

Asahina, Hashimoto, *Ber.*, 1934, **67**, 416.

### Stemonidine

$C_{19}H_{31}O_5N$

MW, 353

Alkaloid isolated from roots of *Stemona japon-*  
*ica*. M.p. 116°.  $[\alpha]_D^{25} = -7.65^\circ$ . Tertiary base.  
 $B, HCl$  : decomp. at 260°.

**Methiodide** : decomp. at 248°.

Suzuki, *J. Pharm. Soc. Japan*, 1929, **49**,  
457; 1939, **59**, 184.

### Stilboestrol.

See Diethylstilboestrol.

See also 4 : 4'-Dihydroxystilbene, and Dodds,  
Goldberg, Lawson, Robinson, *Nature*, 1938, **141**,  
247; Dodds, Lawson, *Nature*, 1937, **139**, 627.

### Styrylamine.

See Cinnamylamine.

### 6-Styryl- $\alpha$ : $\gamma$ -lutidine.

See 4 : 6-Dimethyl- $\alpha$ -stilbazole.

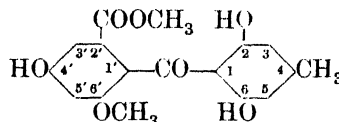
### Suberoxime.

See under Cycloheptanone.

### Succinodiformic Acid.

See 1 : 4-Diketoadipic Acid.

### Sulochrin



$C_{17}H_{16}O_7$

MW, 332

Mycelial constituent of *Oospora sulphurea-*  
*ochracea*. M.p. 262°.

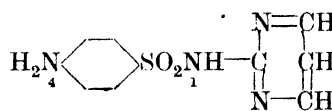
**2 : 4'-Di-Me ether** : m.p. 158°.

**Tri-Me ether** : plates. M.p. 157°.

**Triacetyl** : m.p. 164°.

Nishikawa, *Acta Phytochim.*, 1939, **11**,  
167; *Bulletin of the Agricultural Chemi-*  
*cal Society Japan*, 1940, **16**, 97.

**Sulphadiazine** (*Sulphapyrimidine*, *sulphanil-*  
*amidopyrimidine*,  $N_1$ -2-pyrimidylsulphanilamide)



$C_{10}H_{10}O_2N_4S$

MW, 250

Cryst. from  $H_2O$ . M.p. 255-6° corr. decomp.  
Sol.  $H_2O$  to 12-3 mgm. per c.c. at 37°. Sol.  
acids and alkalis. Na deriv. sol.  $H_2O$  (sol.  $p_H$   
9-6). Used in medicine as bactericide of the  
sulphanilamide class.

$N_4$ -Acetyl : m.p. 258-9° corr. Sol.  $H_2O$  to  
15 mgm. per c.c. at 37°.

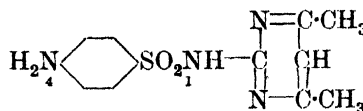
Roblin, Williams, Winnek, English, *J.*  
*Am. Chem. Soc.*, 1940, **62**, 2002.

Roblin, Winnek, English, *J. Am. Chem.*  
*Soc.*, 1942, **64**, 568.

### Sulphaguanidine.

See Sulphanilylguanidine.

**Sulphamethazine** (2-Sulphanilamido-4 : 6-  
dimethylpyrimidine,  $N_1$ -2-[4 : 6-dimethylpyri-  
midyl]-sulphanilamide, *Sulphamezathine*)



$C_{12}H_{14}O_2N_4S$

MW, 278

M.p. 198-9° corr. Sol.  $H_2O$  to 75 mgm. per  
c.c. at 37°. Sol. acids and alkalis. Used in  
medicine as bactericide of the sulphanilamide  
class.



**N<sub>4</sub>-Acetyl**: m.p. 249–50° corr.

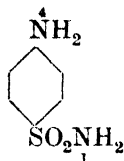
Roblin, Winnek, English, *J. Am. Chem. Soc.*, 1942, **64**, 568.

Caldwell, Kornfeld, Donnell, *J. Am. Chem. Soc.*, 1941, **63**, 2188.

### Sulphamezathine.

See Sulphamethazine.

**Sulphanilamide** (p-Aminobenzenesulphonamide, *Prontosil album*)



**C<sub>6</sub>H<sub>8</sub>O<sub>2</sub>N<sub>2</sub>S**

MW, 172

Leaflets from EtOH.Aq. M.p. 165–6° (163°). Sol. MeOH, EtOH, Et<sub>2</sub>O, Me<sub>2</sub>CO, dil. HCl, hot H<sub>2</sub>O. Sol. 632 parts H<sub>2</sub>O at 0°, 240 parts at 15°, 47.4 parts at 100°. Spar. sol. pet. ether, CHCl<sub>3</sub>. Boil with water, acids or alkalis → sulphanilic acid. Can be diazotised. Extensively employed in medicine in the treatment of streptococcal infections, of meningococcal meningitis, etc., and in the treatment of open wounds to prevent gas gangrene.

**N<sub>1</sub>-Acetyl**: prisms from H<sub>2</sub>O. M.p. 182–4°.

**N<sub>4</sub>-Acetyl**: needles from EtOH.Aq. M.p. 219°.

**N<sub>1</sub>:N<sub>4</sub>-Diacetyl**: prisms from EtOH. M.p. 253.5–255°.

**N<sub>4</sub>-Benzoyl**: cryst. from Py. M.p. 284°.

**N<sub>1</sub>:N<sub>4</sub>-Dibenzoyl**: cryst. from EtOH. M.p. 268–70°.

Carranza, Márques, *Chem. Abstracts*, 1940, **34**, 5422.

Gelmo, *J. prakt. Chem.*, 1908, **77**, 372.

Northey, *Chem. Reviews*, 1940, **27**, 85 (*Bibl.*).

Dewing, Gray, Platt, Stephenson, *J. Chem. Soc.*, 1942, 239.

### Sulphanilamidodimethylpyrimidine.

See Sulphamethazine.

### Sulphanilamidopyridine.

See Sulphapyridine.

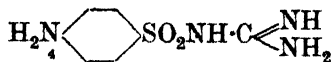
### Sulphanilamidopyrimidine.

See Sulphadiazine.

### Sulphanilamidothiazole.

See Sulphathiazole.

### Sulphanilylguanidine (*Sulphaguanidine*)



**C<sub>7</sub>H<sub>10</sub>O<sub>2</sub>N<sub>4</sub>S**

MW, 214

Cryst. + 1H<sub>2</sub>O from H<sub>2</sub>O. M.p. 142.5–143.5° (sealed tube). M.p. anhyd. 189–90°. Sol. to 10% in boiling H<sub>2</sub>O, 5% in boiling 95% EtOH, 1.5% in boiling acetone. Spar. sol. cold H<sub>2</sub>O, cold EtOH. Sol. dil. min. acids; insol. dil. aq. alkalis. Insol. Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>. Used in medicine as bactericide particularly for bacillary dysentery.

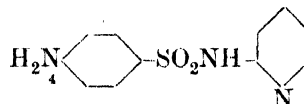
**Hydrochloride**: m.p. 205–6°.

**N<sub>4</sub>-Acetyl**: m.p. 262–6°.

Marshall *et al.*, Bulletin of the Johns Hopkins Hospital, 1940, **67**, 163, (*Chem. Abstracts*, 1940, **34**, 7405).

Dewing, Smith, *Nature*, 1941, **148**, 24.

**Sulphapyridine** (p-Amino-N-2-pyridylbenzenesulphonamide, 2-sulphanilamidopyridine, *M. & B.* 693, *Dagenan*, ω-N-2-pyridylsulphanilamide)



**C<sub>11</sub>H<sub>11</sub>O<sub>2</sub>N<sub>3</sub>S**

MW, 249

Cryst. from EtOH. M.p. 190–1° (191–2°). Sol. H<sub>2</sub>O to 49.5 mgm. per 100 c.c. at 37°. Extensively employed in medicine in the treatment of bacterial infections, especially pneumonia.

**N<sub>4</sub>-Acetyl**: needles from Me<sub>2</sub>CO. M.p. 226–7° (225°).

Márques, *Chem. Abstracts*, 1940, **34**, 5422.

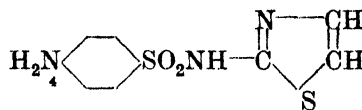
Winterbottom, *J. Am. Chem. Soc.*, 1940, **62**, 160.

Roblin, Winnek, *J. Am. Chem. Soc.*, *ibid.*, 1999.

### Sulphapyrimidine.

See Sulphadiazine.

### Sulphathiazole (2-Sulphanilamidothiazole)



**C<sub>9</sub>H<sub>9</sub>O<sub>2</sub>N<sub>3</sub>S<sub>2</sub>**

MW, 255

Cryst. powder from 45% EtOH. M.p. 202–202.5°. Sol. H<sub>2</sub>O to 60 mgm. per 100 c.c. at 26° (*p<sub>H</sub>* 6.03), 94 mgm. at 37°. Sol. EtOH to 525 mgm. per 100 c.c. at 26°. Used in medicine as a bactericide particularly in treatment of streptococcal and staphylococcal infections, pneumonia and gonorrhea.

**Hydrochloride**: m.p. 193–7°.

*Na deriv.*: m.p. 264.5–265°. *p<sub>H</sub>* of 2% aq. sol. 9.57.

$N_4$ -Acetyl: m.p. 256-7°.

Lott, Bergheim, *J. Am. Chem. Soc.*, 1939, **61**, 3592.

Fosbinder, Walter, *ibid.*, 2032.

### Sulphido- $\alpha$ -alanine.

See Lanthionine.

### Sumaresinolic Acid

$C_{31}H_{50}O_4$  MW, 486

Isolated from Sumatra benzoin. Needles from EtOH.Aq. M.p. 298-9°.  $[\alpha]_D^{24} + 102.2^\circ$  in  $CHCl_3$ .

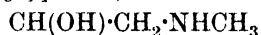
Me ester: cryst. from MeOH.Aq. M.p. 220-1°.  $[\alpha]_D^{24} + 53.6^\circ$  in  $CHCl_3$ .

Et ester: needles from EtOH.Aq. M.p. 207-8°.  
Bromolactone: needles. M.p. 252°.  $[\alpha]_D^{24} + 60.2^\circ$  in  $CHCl_3$ .

Winterstein, Egli, *Z. physiol. Chem.*, 1931, **202**, 207.

Zinke, Liebe, *Monatsh.*, 1918, **39**, 219.

**Sympathol** (1-[p-Hydroxyphenyl]-N-methylaminoethyl alcohol, synephrine, p-[ $\alpha$ -hydroxy- $\beta$ -methylaminoethyl]-phenol)



$C_9H_{13}O_2N$  MW, 167

M.p. 184-5° decomp. Spar. sol. org. solvents.  
B.HCl: cryst. powder. M.p. 149.5-151°.  
 $FeCl_3 \rightarrow$  violet col. Phosphotungstic acid  $\rightarrow$  blue col. Vasoconstrictor. Substitute for epinephrine.

Dibenzoyl: m.p. 176°.

Priestley, Moness, *J. Org. Chem.*, 1940, **5**, 355.

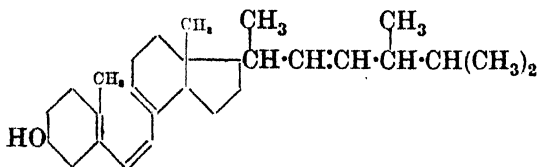
Gordon, *Chem. Zentr.*, 1931, II, 3016.

### Synephrine.

See Sympathol.

## T

### Tachysterol



$C_{29}H_{44}O$  MW, 396

B.p. 220°/high vac. Sol. most org. solvents.  
 $[\alpha]_D^{18} - 70^\circ (-11.5^\circ)$  in  $C_6H_6$ . Absorption maxi-

mum at 2800 Å. with inflexions at 2940 and 2680 Å. Irradiation  $\rightarrow$  calciferol (vitamin  $D_2$ ).

3:5-Dinitro-p-toluy: cryst. from  $Me_2CO$ -MeOH or  $Et_2O$ -MeOH. M.p. 154.5°.  $[\alpha]_D^{18} + 40.4^\circ$  in  $CHCl_3$ .

Citraconic anhydride adduct of acetyl: needles from AcOH.Aq. M.p. 161-2°.  $[\alpha]_D^{20} + 75.2^\circ$  in  $CHCl_3$ .

Windaus, Deppe, Wunderlich, *Ann.*, 1938, **533**, 118.

Windaus, Werder, Luttringhaus, Fernholz, *Ann.*, 1932, **499**, 188.

Lettré, *Ann.*, 1934, **511**, 280.

Grundmann, *Z. physiol. Chem.*, 1938, **252**, 151.

### Taraxasterol

$C_{30}H_{50}O$  MW, 426

Triterpene alcohol from roots of *Taraxacum officinale* and flowers of *Anthemis nobilis*. Needles from EtOH. M.p. 221-2°.  $[\alpha]_D^{17} + 95.9^\circ$  in  $CHCl_3$ .

Acetyl: plates from AcOEt. M.p. 251-2°.  $[\alpha]_D^{18} + 100.5^\circ$  in  $CHCl_3$ .

Bromoacetyl: needles from AcOEt. M.p. 233-4°.

Benzoyl: needles from  $Me_2CO$  or  $C_6H_6$ -EtOH. M.p. 240-1°.  $[\alpha]_D^{12} + 106.8^\circ$  in  $CHCl_3$ .

p-Nitrobenzoyl: needles from  $CHCl_3$ -EtOH. M.p. 277-8°.  $[\alpha]_D^{17} + 98.3^\circ$  in  $CHCl_3$ .

Burrows, Simpson, *J. Chem. Soc.*, 1938, **2042**.

Power, Browning, *J. Chem. Soc.*, 1912, **101**, 2411.

### Taraxerol

$C_{30}H_{50}O$  MW, 426

Triterpene alcohol from rhizomes of *Taraxacum officinale*. Plates from  $CHCl_3$ -EtOH. M.p. 269-71°.  $[\alpha]_D^{18} + 8.4^\circ$  in  $CHCl_3$ .

Acetyl: needles from  $C_6H_6$ -EtOH. M.p. 296-7°.  $[\alpha]_D^{18} + 8.4^\circ$ .

Benzoyl: needles from  $CHCl_3$ -EtOH. M.p. 282-4°.  $[\alpha]_D^{11} + 35.0^\circ$  in  $CHCl_3$ .

Burrows, Simpson, *J. Chem. Soc.*, 1938, **2042**.

### Taraxol

$C_{30}H_{46}O_3$  MW, 454

Triterpene alcohol from rhizomes of *Taraxacum officinale*. Needles from  $C_6H_6$ -EtOH. M.p. above 360°.  $[\alpha]_D^{14} + 78.6^\circ$  in  $CHCl_3$ .

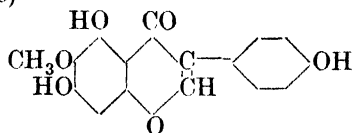
Monoacetyl: plates from  $C_6H_6$ . M.p. 299-301° decomp.  $[\alpha]_D^{14} + 93.9^\circ$  in  $CHCl_3$ .

## Tectorigenin

*Oxide*: needles from MeOH.Aq. M.p. 261–261.5°. *Acetyl*: plates from C<sub>6</sub>H<sub>6</sub>. M.p. 294–7°.

Burrows, Simpson, *J. Chem. Soc.*, 1938, 2042.

**Tectorigenin (5 : 7 : 4'-Trihydroxy-6-methoxyisoflavone)**



C<sub>16</sub>H<sub>12</sub>O<sub>6</sub> MW, 300

Occurs as glycoside, tectoridin, in the rhizomes of *Balameanda chinensis* and *Iris tectorum*, Maxim. Yellow plates from EtOH. M.p. 227° decomp.

*Triacetyl*: prisms. M.p. 187°.

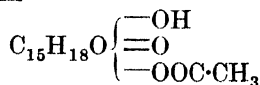
*Tribenzoyl*: plates. M.p. 238°.

*Di-Me ether*: C<sub>18</sub>H<sub>16</sub>O<sub>6</sub>. MW, 328. M.p. 188°. *Acetyl*: m.p. 213–14°.

Asahina, Shibata, Ogawa, *J. Pharm. Soc. Japan*, 1928, 48, 1087.

Shibata, *Chem. Zentr.*, 1927, II, 839.

## Tenulin



C<sub>17</sub>H<sub>22</sub>O<sub>5</sub> MW, 306

Constituent of *Helenium tenuifolium*, *H. elegans*, etc. Cryst. from H<sub>2</sub>O. M.p. 193–5°.  $[\alpha]_D^{20}$  – 21.7° in EtOH. Fish poison. Mild sternutatory action. H(+ Pt) → dihydrotenulin, m.p. 182°. Br → dibromotenulin, m.p. 124–5°. Alkalis → isotenulin, m.p. 162°.

Clark, *J. Am. Chem. Soc.*, 1940, 62, 597; 1939, 61, 1836.

## Testosterone

See Δ<sup>4</sup>-Androstene-17-*cis*-ol-3-one and Δ<sup>5</sup>-Androstene-17-*trans*-ol-3-one.

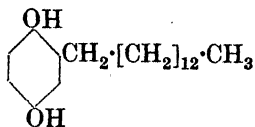
## Tetradecanone-2.

See Methyl dodecyl Ketone.

## 4-Tetradecylenic Acid.

See Tsuzuic Acid.

**Tetradecylhydroquinone (Myristylquinol, 2 : 5-dihydroxytetradecylbenzene)**



C<sub>20</sub>H<sub>34</sub>O<sub>2</sub> MW, 306

Cryst. from pet. ether. M.p. 110°. Ag<sub>2</sub>O → tetradecylbenzoquinone, m.p. 77.5°.

## 970 1 : 4 : 5 : 7-Tetrahydroxy-2-methyl-anthraquinone

*Di-Me ether*: 2 : 5-dimethoxytetradecylbenzene. B.p. 165°/0.5 mm.

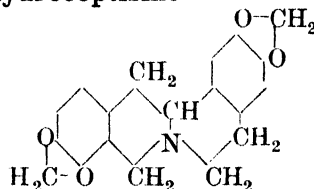
*Di-Et ether*: 2 : 5-diethoxytetradecylbenzene. B.p. 183°/0.1 mm.

Cook, Heilbron, Lewis, *J. Chem. Soc.*, 1942, 660.

## Tetrahydroanisole.

See under Cyclohexenol.

## Tetrahydrocoptisine



C<sub>19</sub>H<sub>17</sub>O<sub>4</sub>N MW, 323

l-. *Corydalis-D*.

Occurs in Chinese *Corydalis ambigua*. Prisms. M.p. 204°.  $[\alpha]_D^{25}$  – 295°.

*B, HCl*: m.p. about 250°.

*B, HBr*: m.p. about 260°.

dl-. *Corydalis-E*.

From Chinese *Corydalis ambigua*. Needles from CHCl<sub>3</sub>. M.p. 222–3°. B.p. 260°/0.01 mm.

*B, HCl*: m.p. 246°.

Späth, Posega, *Ber.*, 1929, 62, 1032.

Huang-Minlon, *Ber.*, 1936, 69, 1737.

Chou, *Chinese Journal of Physiology*, 1936, 10, 507, (*Chem. Abstracts*, 1937, 31, 1161).

## Tetrahydrocuminic Acid.

See Phellandric Acid.

## Tetrahydrodiphenyl.

See Phenylcyclohexene.

## Tetrahydrogeraniol.

See 3 : 7-Dimethyloctanol-1.

## Tetrahydrolinalool.

See 3 : 7-Dimethyloctanol-3.

## Tetrahydrophenetole.

See under Cyclohexenol.

## Tetrahydrophenylacetaldehyde.

See Cyclohexenylacetaldehyde.

## Tetrahydrophenylacetone.

See Cyclohexenylacetone.

## Tetrahydrophenylpropionic Acid.

See Cyclohexenylpropionic Acid.

## 3 : 7 : 3' : 4'-Tetrahydroxyflavone.

See Fustin.

## 5 : 7 : 3' : 4'-Tetrahydroxyisoflavone.

See Orobol.

## 1 : 4 : 5 : 7 - Tetrahydroxy - 2 - methyl - anthraquinone.

See Catenarin.

**1 : 4 : 5 : 8 - Tetrahydroxy - 2 - methyl - anthraquinone.**

See Cynodontin.

**3 : 4 : 3' : 5' - Tetrahydroxystilbene 4 - methyl Ether.**

See Rhapontigenin.

**Tetramethyldiaminobenzaldehyde.**

See under 2 : 4-Diaminobenzaldehyde.

**Tetramethylphenol.**

See Durenol and Prehnitenol.

**Tetramethyltetraethylporphin.**

See Aetioporphyryn.

**Thiamin.**

See Aneurin.

**Thioamyl Alcohol.**

See Amyl Mercaptan.

**Thiocarbanilic Acid.**

See Phenylthiocarbamic Acid.

**Thiodiphenylamine.**

S-Dioxide : see Diphenylamine sulphone.

**Thiododecyl Alcohol.**

See Dodecyl Mercaptan.

**Thioethylene Glycol.**

See Ethylene Thioglycol.

**Thioglycol.**

See Ethylene Thioglycol.

**Thiophenetidine.**See under *p*-Aminothiophenol.**Thiotoluidine.**

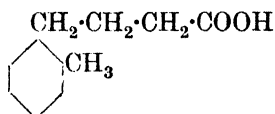
See Diaminoditolyl sulphide.

**Toluy catechol.**

See Dihydroxymethylbenzophenone.

**Toluy resorcinol.**

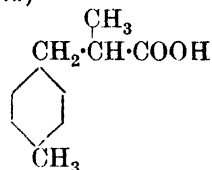
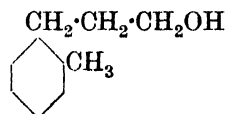
See Dihydroxymethylbenzophenone.

**3-*o*-Tolylbutyric Acid** $\text{C}_{11}\text{H}_{14}\text{O}_2$  MW, 178

M.p. 70.5°.

Et ester :  $\text{C}_{13}\text{H}_{18}\text{O}_2$ . MW, 206. B.p. 140–50°/9 mm.Wessely, Wang, *Ber.*, 1940, 73, 19.**2-*p*-Tolylbutyric Acid.**

See Curcumatic Acid.

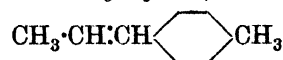
**2 : 4-Tolylenediamine.**2-*N*-Phenyl : see 5-Amino-2-methyldiphenylamine.4-*N*-Phenyl : see 3-Amino-4-methyldiphenylamine.**2 : 5-Tolylenediamine.**2-*N*-Phenyl : see 4-Amino-2-methyldiphenylamine.5-*N*-Phenyl : see 4-Amino-3-methyldiphenylamine.**3 : 4-Tolylenediamine.**3-*N*-Phenyl : see 6-Amino-3-methyldiphenylamine.4-*N*-Phenyl : see 2-Amino-4-methyldiphenylamine.**2-*p*-Tolylisobutyric Acid** ( $\alpha$  : 4-Dimethylhydrocinnamic acid) $\text{C}_{11}\text{H}_{14}\text{O}_2$  MW, 178Plates from  $\text{H}_2\text{O}$ . M.p. 85°. B.p. 168–9°/9 mm.Et ester :  $\text{C}_{13}\text{H}_{18}\text{O}_2$ . MW, 206. B.p. 130–2°/10 mm.Amide :  $\text{C}_{11}\text{H}_{15}\text{ON}$ . MW, 177. Needles from  $\text{H}_2\text{O}$ . M.p. 130°.Ruzicka, Ehmann, Delbes, *Helv. Chim. Acta*, 1932, 15, 162.***m*-Tolylisopropyl Alcohol.**See Methyl-*m*-xylylcarbinol.**3-*o*-Tolylpropyl Alcohol** ( $\gamma$ -Hydroxy-*o*-propyltoluene) $\text{C}_{10}\text{H}_{14}\text{O}$  MW, 150

B.p. 136°/15 mm.

Phenylurethane : needles from pet. ether. M.p. 58°.

Harvey, Heilbron, Wilkinson, *J. Chem. Soc.*, 1930, 428.**3-*m*-Tolylpropyl Alcohol** ( $\gamma$ -Hydroxy-*m*-propyltoluene).B.p. 140°/14 mm.  $D_4^{20}$  0.9609.

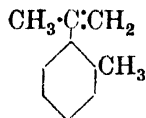
Acetyl : b.p. 136°/10 mm.

v. Braun, Grabowski, Kirschbaum, *Ber.*, 1913, 46, 1274.**1-*p*-Tolylpropylene** (1-Methyl-4-propenylbenzene,  $\beta$  : 4-dimethylstyrene) $\text{C}_{10}\text{H}_{12}$  MW, 132B.p. 195–7°, 92–3°/20 mm., 83–5°/10 mm.  $D^{18}$  0.9057.

Nitroschloride : needles. M.p. 135°.

Klages, *Ber.*, 1902, 35, 2254.

**2-o-Tolylpropylene** (1-Methyl-2-isopropenylbenzene, o-isopropenyltoluene,  $\alpha$ : 2-dimethylstyrene)

C<sub>10</sub>H<sub>12</sub>

MW, 132

B.p. 172-3°. D<sub>20</sub> 0.9076. Oxidises in air.Tiffeneau, *Ann. chim.*, 1907, 10, 194.

**2-m-Tolylpropylene** (1-Methyl-3-isopropenylbenzene, m-isopropenyltoluene,  $\alpha$ : 3-dimethylstyrene).

B.p. 185-6°. D<sub>20</sub> 0.9115. Oxidises in air.

See previous reference.

**2-p-Tolylpropylene** (1-Methyl-4-isopropenylbenzene, p-isopropenyltoluene,  $\alpha$ : 4-dimethylstyrene).

M.p. — 20°. B.p. 184-5°. D<sub>20</sub> 0.9122. Oxidises in air. Slowly polymerises.

Nitroschloride: cryst. from MeOH. M.p. 100-2°.

Tiffeneau, *Ann. chim.*, 1907, 10, 194.Perkin, Pickles, *J. Chem. Soc.*, 1905, 87, 653.**Torulin.**

See Aneurin.

**Totarol**C<sub>20</sub>H<sub>30</sub>O

MW, 286

Diterpene alcohol occurring in *Podocarpus totara*. Tricyclic and containing 3 ethylenic linkages. Needles from ligroin. M.p. 132°. [ $\alpha$ ]<sub>D</sub><sup>20</sup> + 41.3° in EtOH. Heat with Se  $\rightarrow$  7-hydroxy-1-methylphenanthrene and 1-methylphenanthrene.

Formyl: hexagonal prisms from EtOH. M.p. 125.5°.

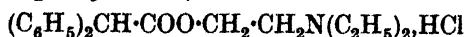
Acetyl: rectangular plates from EtOH. M.p. 121.5°. [ $\alpha$ ]<sub>D</sub><sup>18</sup> + 44.6° in Et<sub>2</sub>O.

Hydrogen phthaloyl: prisms from EtOH. M.p. 161-3°.

Me ether: cryst. from CHCl<sub>3</sub>-EtOH. M.p. 92-92.5°. [ $\alpha$ ]<sub>D</sub><sup>18</sup> + 41.95° in Et<sub>2</sub>O.Short, Stromberg, *J. Chem. Soc.*, 1937, 516.**T. P. N.**

See Coenzyme II.

**Trasentin** (Hydrochloride of 2-diethylaminoethyl diphenylacetate)

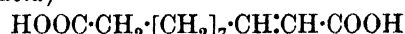
C<sub>20</sub>H<sub>26</sub>O<sub>2</sub>NCl

MW, 347.5

Cryst. M.p. 113-14°. Sol. H<sub>2</sub>O. Parasympathetic antispasmodic.

Ciba, Swiss Ps., 190,541, 192,070, 192,697, (*Chem. Abstracts*, 1938, 32, 589, 4174, 4284).

**Traumatic Acid** (1-Decylene-1:10-dicarboxylic acid)

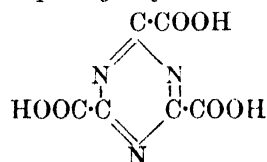
C<sub>12</sub>H<sub>20</sub>O<sub>4</sub>

MW, 228

Plant wound hormone isolated from string bean pods. Cryst. from EtOH or Me<sub>2</sub>CO. M.p. 165-6°.

English, Benner, Haagen-Smit, *J. Am. Chem. Soc.*, 1939, 61, 3434.

**Triazine-tricarboxylic Acid** (Cyanuric tri-carboxylic acid, paracyanofornic acid)

C<sub>6</sub>H<sub>3</sub>O<sub>6</sub>N<sub>3</sub>

MW, 213

Cryst. powder. M.p. above 250° decomp. Spar. sol. H<sub>2</sub>O. Insol. EtOH, Et<sub>2</sub>O. Boiling H<sub>2</sub>O  $\rightarrow$  NH<sub>3</sub> + oxalic acid.

Tri-Me ester: C<sub>9</sub>H<sub>9</sub>O<sub>6</sub>N<sub>3</sub>. MW, 252. Needles from EtOH. M.p. 154°.

Tri-Et ester: C<sub>12</sub>H<sub>15</sub>O<sub>6</sub>N<sub>3</sub>. MW, 297. Needles from EtOH. M.p. 165°.

Tri-isobutyl ester: needles from EtOH. M.p. 158°.

Tri-Me-amide: needles from EtOH or H<sub>2</sub>O. M.p. above 250° with decomp.

Trinitrile: C<sub>6</sub>N<sub>6</sub>. MW, 156. M.p. 119° with sublimation. B.p. 262°.

Weddige, *J. prakt. Chem.*, 1874, 10, 212. $\alpha$ :  $\beta$ : 4-Tribromodiphenylethane.

See under p-Bromostilbene.

**Tricetylamine.**

See under Cetylamine.

**1:1:1-Trichloro-tert.-butyl Alcohol.**

See Chloretone.

 $\psi$ -Trichloro- $\gamma$ -hydroxybutyrophenone.

See Chloralacetophenone.

**5:5:5-Trichloro-4-hydroxy-2-ketopentane.**

See Chloralacetophenone.

**2:2':2''-Trichlorotriethylamine.**

See under 2-Chloroethylamine.

**Tridecanol.**

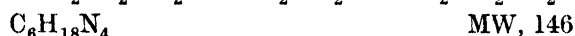
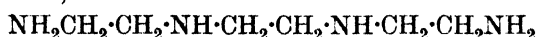
See Di-n-hexylcarbinol.

**Tridecanone-7.**

See Di-n-hexyl Ketone.

**4-Tridecylene-1-carboxylic Acid.**

See Tsuzuic Acid.

**Triethylenetetramine** (N:N'-Di-[2-aminoethyl]-ethylenediamine, 1:2-di-[aminoethylamino]-ethane)

B.p. 174°/31 mm., 157°/20 mm.

B<sub>4</sub>HCl: needles from EtOH-HCl. M.p. 266-70°.Hydrogen oxalyl: needles from H<sub>2</sub>O. Decomp. at 243° corr.Tetrabenzoyl: micro-cryst. from CHCl<sub>3</sub>-EtOH. M.p. 238°.Tetrapicrate: prisms from H<sub>2</sub>O. Decomp. at 240° corr.Peacock, *J. Chem. Soc.*, 1936, 1518.van Alphen, *Rec. trav. chim.*, 1936, 55, 412.Fargher, *J. Chem. Soc.*, 1920, 117, 1351.**Triglycine.**

See Diglycylglycine.

**α : 3 : 4-Trihydroxyacetophenone.**

See 3 : 4-Dihydroxyphenacyl Alcohol.

**4 : 5 : 7 - Trihydroxyanthraquinone - 2 - carboxylic Acid.**

See Emodic Acid.

**Trihydroxybutyric Acid.**

See Erythronic Acid and Threonic Acid.

**7 : 3' : 4'-Trihydroxyflavanol.**

See Fustin.

**1 : 6 : 8-Trihydroxy-3-hydroxymethyl-anthraquinone.**

Me ether: see Carviolin.

**5 : 7 : 4' - Trihydroxy - 6 - methoxyiso - flavone.**

See Tectorigenin.

**8 : 9 : 15-Trihydroxypalmitic Acid.**

See Aleuritic Acid.

**3 : 5 : 4'-Trihydroxystilbene.**

See Resveratrol.

**Triketohydrindene Hydrate.**

See Ninhydrin.

**Trillin (3-α-Glucoside of diosgenin)**Occurs in *Trillium erectum* as the diglucoside, trillarin, m.p. 197-200°. Cryst. +  $\frac{1}{2}\text{H}_2\text{O}$  from MeOH. M.p. 275-80°. EtOH-HCl → diosgenin.

Tetra-acetyl: cryst. from MeOH. M.p. 197°.

Marker, Krueger, *J. Am. Chem. Soc.*, 1940, 62, 2548, 3349.**2 : 4 : 6-Trimethoxyisobutyrophenone.**

See Conglomerone.

**Trimethylallantoin.**

See Caffoline.

**1 : 1 : 1-Trimethylbutane.**

See 2 : 2-Dimethylpentane.

**Trimethylbutylene.**

See Dimethylpentene.

**Trimethylcaffolide.**

See under Caffolide.

**4 : 5 : 5-Trimethylcyclopentene-1-carboxylic Acid.**See Δ<sup>5</sup>-Campholytic Acid.**Trimethylene chloriodide.**

See 3-Chloro-1-iodopropane.

**Trimethylhydroquinone.**

See ψ-Cumohydroquinone.

**Trimethylresorcinol.**

See Dihydroxy-ψ-cumene and Dihydroxyhemimellitene.

**Trimethyltetrolic Acid.**

See tert.-Butylpropionic Acid.

**Trinitrotrimethylenetriamine.**

See Cyclonite.

**Triphenylchloroethylene.**

See Chlorotriphenylethylene.

**Triphenylmethylaniline.**

See α-Aminotriphenylmethane.

**α-Tritisterol**Occurs in unsaponifiable portion of wheat germ oil. Needles from Me<sub>2</sub>CO-MeOH. M.p. 114-15°. [α]<sub>D</sub><sup>20</sup> + 54.3° in EtOH.Acetyl: leaflets from EtOH. M.p. 107-8°. [α]<sub>D</sub><sup>20</sup> + 70.4° in CHCl<sub>3</sub>.

3 : 5-Dinitrobenzoyl: cryst. from AcOEt. M.p. 182°.

β-Naphthoyl: needles. M.p. 158-9°.

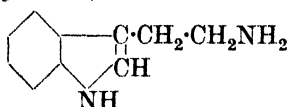
Karrer, Salomon, *Helv. Chim. Acta*, 1937, 20, 424.Karrer, Salomon, Fritzsche, *ibid.*, 1422.Todd, Bergel, Waldmann, Work, *Biochem. J.*, 1937, 31, 2247.Drummond, Hoover, *ibid.*, 1852.**β-Tritisterol**Occurs in unsaponifiable portion of wheat germ oil. Needles from MeOH. M.p. 97°. [α]<sub>D</sub><sup>20</sup> + 49.2° in EtOH.Acetyl: needles from MeOH-EtOH. [α]<sub>D</sub><sup>20</sup> + 55.5° in CHCl<sub>3</sub>.Acetyl-dibromide: cryst. from Me<sub>2</sub>CO. M.p. 160-2°.

See previous references.

**Trypaflavine.**

See Acriflavine.

**Tryptamine** (3-[ $\omega$ -Aminoethyl]-indole, 2-[3-indolyl]-ethylamine)



$C_{10}H_{12}N_2$

MW, 160

Needles from ligroin. M.p.  $146^\circ$  ( $115^\circ$ ). Sol. EtOH,  $Me_2CO$ . Prac. insol.  $H_2O$ ,  $Et_2O$ ,  $CHCl_3$ ,  $C_6H_6$ .

$B \cdot HCl$ : prisms. M.p.  $248-9^\circ$ .

$B_2 \cdot (CH_2 \cdot COOH)_2$ : needles from AcOEt. M.p.  $162^\circ$  decomp.

$B_2 \cdot (CH_2 \cdot COOH)_2$ : m.p.  $201^\circ$ .

$B_2 \cdot (CH_2 \cdot CH_2 \cdot COOH)_2$ : needles from AcOEt. M.p.  $203^\circ$  decomp.

$N$ -Thioformyl: plates from  $CHCl_3$ -pet. ether. M.p.  $82^\circ$ .

$N$ -Acetyl: cryst. from pet. ether. M.p.  $77^\circ$ .

$N$ -Benzoyl: prisms. M.p.  $137-8^\circ$ .

$N$ -Me: see 3-[ $\omega$ -Methylaminoethyl]-indole.

Picrate: dark red prisms from  $Et_2O$ . M.p.  $247^\circ$  decomp.

Majima, Hoskins, *Ber.*, 1925, **58**, 2045.

Manske, *J. Am. Chem. Soc.*, 1929, **51**, 1202.

Späth, Lederer, *Ber.*, 1930, **63**, 123.

**Tsuzuic Acid** (4-Tetradecenoic acid, 4-tetradecylenic acid, 4-tridecylene-1-carboxylic acid)



$C_{14}H_{26}O_2$

MW, 226

Occurs in tohaku oil (seed oil of *Lindera obtusiloba*) and in seed oil of "tsuzu" (*Tetradenia glauca*). M.p.  $18-18.5^\circ$ . B.p.  $185-8^\circ/13$  mm.  $D_4^{20}$  0.9024.  $n_D^{20}$  1.4559.

Me ester:  $C_{15}H_{28}O_2$ . MW, 240. B.p.  $158-60^\circ/15$  mm.  $D_4^{15}$  0.8857.  $n_D^{15}$  1.4489.

Et ester:  $C_{16}H_{30}O_2$ . MW, 254. B.p.  $168-70^\circ/15$  mm.  $D_4^{15}$  0.8783.  $n_D^{15}$  1.4479.

p-Bromophenacyl ester: m.p.  $61.3^\circ$ .

p-Phenylphenacyl ester: m.p.  $54.5^\circ$ .

Toyama, *J. Soc. Chem. Ind. Japan*, 1937, **40**, 285.

Komori, Ueno, *Bull. Chem. Soc. Japan*, 1937, **12**, 433.

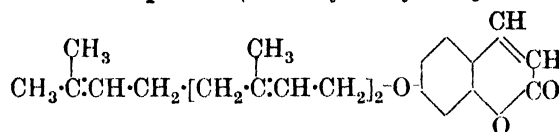
Tsujimoto, *Chem. Abstracts*, 1928, **22**, 4470.

## U

**Umbelliferone.**

d-Glucoside: see Skimmin.

**Umbelliprenin** (Umbelliferone farnesyl ether)



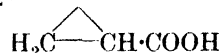
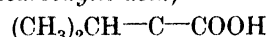
$C_{24}H_{30}O_3$

MW, 246

Occurs in angelica seed (*Angelica archangelica*). Cryst. from  $Et_2O$ -pet. ether. M.p.  $61-3^\circ$ . Cold  $AcOH-H_2SO_4 \rightarrow$  umbelliferone.

Späth, Vierhapper, *Ber.*, 1938, **71**, 1667; *Monatsh.*, 1938, **72**, 179.

**Umbellularic Acid** (1-Isopropylcyclopropane-1:2-dicarboxylic acid)



$C_8H_{12}O_4$

MW, 172

*Cis*-.

*dl*-.

Needles from  $C_6H_6$ -pet. ether. M.p.  $126-7^\circ$ . Also labile form; m.p.  $117-18^\circ$ .

p-Phenylphenacyl ester: needles from AcOH. M.p.  $116-17^\circ$ .

Anhydride: b.p.  $140^\circ/20$  mm.

*d*-.

Needles +  $1H_2O$  from  $H_2O$ , m.p.  $78-9^\circ$ . Prisms from  $C_6H_6$ -pet. ether, m.p.  $119-20^\circ$ .  $[\alpha]_D^{20} + 86.9^\circ$  in  $CHCl_3$ .

Acid brucine salt: plates from  $H_2O$ . M.p.  $110-20^\circ$ .  $[\alpha]_{5461}^{20} + 6.2^\circ$  in  $Me_2CO$ .

*l*-.

Needles +  $1H_2O$  from  $H_2O$ , m.p.  $78-9^\circ$ . Prisms from  $C_6H_6$ -pet. ether, m.p.  $119-20^\circ$ .  $[\alpha]_D^{15} - 88.8^\circ$  in  $CHCl_3$ .

Acid brucine salt: needles from  $H_2O$ .  $[\alpha]_{5461}^{16} - 29.5^\circ$  in  $Me_2CO$ .

*Trans*-.

*dl*-.

Prisms from  $H_2O$ . M.p.  $197^\circ$ . Sol.  $Me_2CO$ , AcOEt. Spar. sol.  $C_6H_6$ ,  $CHCl_3$ . Heat alone or with  $CH_3COCl$  or  $Ac_2O \rightarrow$  *cis*-anhydride.

p-Phenylphenacyl ester: needles from AcOEt or  $Me_2CO$ . M.p.  $175-6^\circ$ .

Di-Et ester: b.p.  $130-5^\circ/20$  mm.

*d*-.

Needles from  $H_2O$ . M.p.  $155^\circ$ .  $[\alpha]_{5461}^{21} + 232.1^\circ$  in  $Me_2CO$ .

Brucine salt: needles from  $H_2O$ .  $[\alpha]_{5461}^{22} - 15.4^\circ$  in  $CHCl_3$ .

*l*-.

Needles from  $H_2O$ . M.p.  $153^\circ$ .  $[\alpha]_{5461}^{21} - 236.2^\circ$  in  $Me_2CO$ .

**Brucine salt**: prisms from  $H_2O$ . M.p. 149–51° decomp.  $[\alpha]_{D}^{25} - 98.8^\circ$  in  $CHCl_3$ .

Phillips, Ramage, Simonsen, *J. Chem. Soc.*, 1936, 828.

Rydon, *ibid.*, 829.

Guha, Muthanna, *Ber.*, 1938, 71, 2668.

#### Undecanol-5.

See Butyl-*n*-hexylcarbinol.

#### Undecanol-6.

See Di-*n*-amylcarbinol.

#### Undecanone-5.

See Butyl *n*-hexyl Ketone.

#### 5-Undecine.

See Butylamylacetylene.

#### 4-Undecylene-1-carboxylic Acid.

See Linderic Acid.

#### *p*-Ureidophenetole.

See Dulcin.

#### Uteroverdin.

See Biliverdin.

### V

#### Vanguerigenin

$C_{30}H_{46}O_3$  MW, 454

Needles +  $\frac{1}{3}$  EtOH from EtOH. M.p. 266°.  $[\alpha]_D^{25} + 191.3^\circ$  in  $CHCl_3$ . Heat at 300°  $\rightarrow$  vanguardol, m.p. 207°.

*Me ester*: cryst. from EtOH. M.p. 195°.

*Acetyl*: prisms from EtOH. M.p. 248°.

*Acetyl*: needles + 1 EtOH from EtOH. M.p. 295°.

*Lactone*: needles from EtOH.Aq. M.p. 281°.

Merz, Tschubel, *Ber.*, 1939, 72, 1017.

#### Vanguerin

$C_{41}H_{64}O_{11}$  MW, 732

From *Vangueria tomentosa*. Powder. Sinters at 255–60°. M.p. about 275–80° decomp. Sol. hot EtOH, Py.  $[\alpha]_D^{25} - 10.1^\circ$  in dioxan.  $Ac_2O-H_2SO_4 \rightarrow$  yellow  $\rightarrow$  red  $\rightarrow$  bluish violet  $\rightarrow$  brown col. Hyd.  $\rightarrow$  vanguardin + rhamnose + arabinose.

*Penta-acetyl*: powder. Decomp. at 184°.

Merz, Tschubel, *Ber.*, 1939, 72, 1017.

#### *o*-Vanillic Acid.

See under 2 : 3-Dihydroxybenzoic Acid.

#### *o*-Vanillin.

See under 2 : 3-Dihydroxybenzaldehyde.

#### Veratramine

$C_{26}H_{35}O_2N$  MW, 393

Alkaloid of white hellebore (*Veratrum grandiflorum*, Loes). Monohydrate, m.p. 209.5–210.5°.  $[\alpha]_D^{19}$  anhyd.  $- 70^\circ$  in MeOH.  $H(+ Pt) \rightarrow$  dihydroveratramine, m.p. 197–8°.

*B,HCl*: plates from EtOH. M.p. 201–2°.

*Monoacetyl*: prisms from EtOH.Aq. M.p. 201–2°.

*Diacetyl*: needles. M.p. 205.5–206°.

*Picrate*: yellow plates from EtOH.Aq. M.p. 217.5–218°.

Saito, *Bull. Chem. Soc. Japan*, 1940, 15, 22.

#### *o*-Veratric Aldehyde.

See under 2 : 3-Dihydroxybenzaldehyde.

#### 2-Veratrolybenzoic Acid.

See under 3' : 4'-Dihydroxybenzophenone-2-carboxylic Acid.

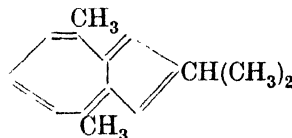
#### Veratrolycarbinol.

See under 3 : 4-Dihydroxyphenacyl Alcohol.

#### Veratrolyformic Acid.

See under 3 : 4-Dihydroxybenzoylformic Acid.

**Vetivazulene** (4 : 8-Dimethyl-2-isopropylazulene)



$C_{15}H_{18}$  MW, 198

B.p. 140–60°/2 mm.

*Picrate*: black needles from EtOH. M.p. 121.5–122°.

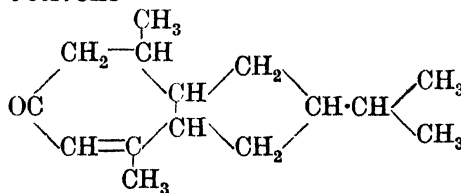
*sym-Trinitrobenzene add. comp.*: black needles with copper reflex from EtOH. M.p. 151.5–152°.

2 : 4 : 6-Trinitrotoluene *add. comp.*: copper coloured needles. M.p. 80.5–81°.

Pfau, Plattner, *Helv. Chim. Acta*, 1939, 22, 202; 1936, 19, 858.

Coates, Cook, *J. Chem. Soc.*, 1942, 559.

#### $\beta$ -Vetivone



$C_{15}H_{22}O$  MW, 218

Isolated from vetiver oil. M.p. 44–45°. B.p. 141–2°/2 mm.  $D_4^{20}$  1.0001.  $n_D^{20}$  1.5309.  $[\alpha]_D - 38.9^\circ$  in EtOH.

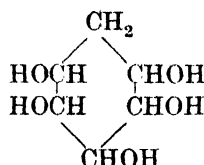
*Semicarbazone*: leaflets from Py. M.p. 288–9°.  $[\alpha]_D - 71.1^\circ$  in AcOH.

2 : 4-Dinitrophenylhydrazone: m.p. 190.5–191°.

Naves, Perrotet, *Helv. Chim. Acta*, 1941, 24, 3.

Pfau, Plattner, *Helv. Chim. Acta*, 1940, 23, 768; 1939, 22, 640.



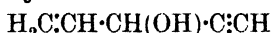
**Viburnitol** (*Pentahydroxycyclohexane*) $C_6H_{12}O_5$ 

MW, 164

Isolated from *Viburnum tinus*, L. Needles +  $H_2O$  from  $H_2O$ . M.p. anhyd.  $180-1^\circ$  corr. Very sol.  $H_2O$ . Spar. sol. EtOH.  $[\alpha]_D - 49.5^\circ$  in  $H_2O$ .

*Penta-acetyl*: needles. M.p.  $125-6^\circ$ .

Hérissé, Poirot, *J. pharm. chim.*, 1937, 26, 385 (*Chem. Abstracts*, 1938, 32, 7024).

**Vinylethynylcarbinol** $C_5H_6O$ 

MW, 82

B.p.  $128.5-129.5^\circ$ ,  $83.5-84.5^\circ/150$  mm.  $n_D^{16} 1.4545$ .

*Acetyl*: b.p.  $87-8^\circ/100$  mm.  $n_D^{25} 1.4319$ .

*Phenylurethane*: needles from pet. ether. M.p.  $37^\circ$ .

$\alpha$ -*Naphthylurethane*: needles from pet. ether. M.p.  $127.5-128.5^\circ$ .

Jones, McCombie, *J. Chem. Soc.*, 1942, 735.

Lespieau, Lombard, *Bull. Soc. chim.*, 1935, 2, 369.

**Vinylidene fluoride.**

See Difluoroethylene.

**Vinylxylene.**

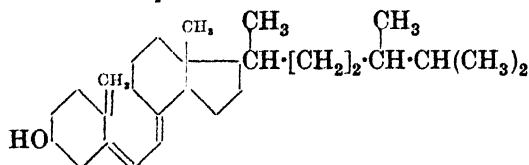
See Dimethylstyrene.

**Vitamin B<sub>1</sub>.**

See Aneurin.

**Vitamin B<sub>6</sub>.**

See Adermin.

**Vitamin D<sub>4</sub>** $C_{28}H_{46}O$ 

MW, 398

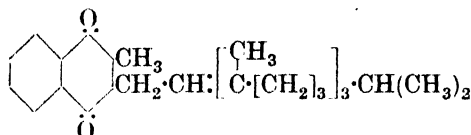
Plates from  $Me_2CO$ . Aq. M.p.  $107-8^\circ$ .  $[\alpha]_D^{18} + 89.3^\circ$  in  $Me_2CO$ . Shows absorption maximum at  $2650 \text{ \AA}$ . Antirachitic activity about one-half that of calciferol on rat tests.

3:5-*Dinitrobenzoyl*: yellow needles. M.p.  $135-6^\circ$ .  $[\alpha]_D^{18} + 94.5^\circ$  in  $Me_2CO$ .

Windaus, Langer, *Ann.*, 1933, 508, 105.

Windaus, Trautmann, *Z. physiol. Chem.*, 1937, 247, 185.

Windaus, Güntzel, *Ann.*, 1939, 538, 120.

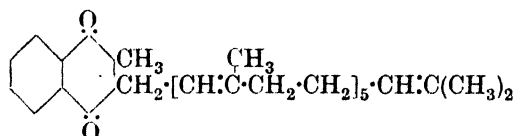
**Vitamin K<sub>1</sub>** (2-Methyl-3-phytyl-1:4-naphthoquinone) $C_{31}H_{46}O_2$ 

MW, 450

Fat soluble dietary factor essential for blood coagulation. Widely distributed in green leaves and vegetables, especially chestnut leaves and alfalfa. Lemon yellow oil which separates from  $Me_2CO$  or EtOH at  $-70^\circ$  in pale yellow rosettes, m.p.  $-20^\circ$ . Sol. EtOH,  $Me_2CO$ , hexane,  $C_6H_6$ ,  $CHCl_3$ . Spar. sol. MeOH. Insol.  $H_2O$ . NaOEt  $\rightarrow$  blue col. Absorption maxima at 2400, 2480 (main), 2610, 2700 and 3230  $\text{\AA}$ . Reductive acetylation  $\rightarrow$  diacetyl-dihydro-comp., m.p.  $62-3^\circ$ . Reductive benzylation  $\rightarrow$  dibenzoyl-dihydro-comp., m.p.  $85-6^\circ$ .  $CrO_3 \rightarrow$  phthalic acid + 2-methyl-1:4-naphthoquinone-3-acetic acid.

Doisy, Binkley, Thayer, *Chemical Reviews*, 1941, 28, 477, (*Review and Bibl.*).

Fieser, *J. Am. Chem. Soc.*, 1939, 61, 3467.

**Vitamin K<sub>2</sub>**

Probable structure

 $C_{41}H_{56}O_2$ 

MW, 580

Fat soluble dietary factor effective in controlling blood coagulation. Widely distributed in green leaves and vegetables. Isolated most readily from chestnut leaves and putrefied fish meal. Yellow cryst. from  $Me_2CO$ -EtOH or MeOH- $CHCl_3$ . M.p.  $51-2^\circ$ . Slightly less sol. in org. solvents than vitamin K<sub>1</sub>. Absorption spectrum similar to that of K<sub>1</sub>. Reductive acetylation  $\rightarrow$  diacetyl-dihydro-comp., m.p.  $59.5-60^\circ$ .

Binkley, McKee, Thayer, Doisy, *J. Biol. Chem.*, 1940, 133, 721.

Doisy, Binkley, Thayer, *Chemical Reviews*, 1941, 28, 477 (*Review and Bibl.*).

**Vitexin** $C_{15}H_{14}O_7$ 

MW, 306

Pigment from wood of *Vitex littoralis* or *Saponaria officinalis*. Yellow micro-plates from 40% Py. M.p. 263°.

*Acetyl deriv.*: colourless prismatic needles from AcOH. M.p. 257–8°.

Perkin, *J. Chem. Soc.*, 1898, **73**, 1019.

Barger, *J. Chem. Soc.*, 1906, **89**, 1210.

Péteri, *J. Chem. Soc.*, 1939, 1635.

#### Voluntal.

See under Carbamic Acid.

H<sub>2</sub>O. Readily sol. dil. NH<sub>4</sub>OH and NaOH to yellow sols., and in 2*N*/HCl to colourless sol. H<sub>2</sub>O<sub>2</sub> → leucopterin.

Wieland, Purrmann, *Ann.*, 1940, **544**, 163.

Schöpf, Kottler, *Ann.*, 1939, **539**, 128.

Schöpf, Becker, *Ann.*, 1936, **524**, 55, 126; 1933, **507**, 266.

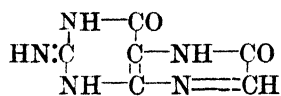
Purrmann, *Ann.*, 1941, **548**, 284.

#### Xeronic Acid.

See Diethylmaleic Acid.

### X

#### Xanthopterin



C<sub>6</sub>H<sub>5</sub>O<sub>2</sub>N<sub>5</sub>

MW, 179

Yellow wing pigment of butterflies, e.g. *Gonepteryx rhamni*, *Appias nero*, *Colias edusa*, etc. Amorph. hygroscopic mass. M.p. above 410° but carbonises above 360°. Prac. insol.

### Z

#### Zeorin

C<sub>30</sub>H<sub>52</sub>O<sub>2</sub>

MW, 444

Triterpene from various lichens, e.g. *Anaptychia speciosa*, *A. hypoleuca*, *A. heterochroa*. Hexagonal pyramids from MeOH. M.p. 253°.  $[\alpha]_D^{24} + 101.4^\circ$  in Py.

*Acetyl deriv.*: cryst. powder from AcOH.Aq. M.p. 178°.

Asahina, Yosioka, *Ber.*, 1940, **73**, 742.

Asahina, Akagi, *Ber.*, 1938, **71**, 980.

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